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CONTENTS

PROCEEDINGS, PART I

PAGE

Summary of the Proceedings of the Fortieth Annual Meeting 1
Fuels of Today and Tomorrow: Annual Address by the President—Arno C. Fieldner. 31
Annual Report of the Executive Committee 54

COMMITTEE REPORTS

FERROUS METALS

Report of Committee A-1 on Steel 81
Report of Research Committee on Yield Point of Structural Steel 87
Discussion 101
Report of Committee A-2 on Wrought Iron 103
Appendix. Proposed Revisions in Tentative Standards for Wrought Iron 107
Report of Committee A-3 on Cast Iron 109
Report of Committee A-5 on Corrosion of Iron and Steel 111
Report of the Wire Test Committee 117
Appendix to the Report of the Wire Test Committee 122
Report of Committee A-7 on Malleable Iron Castings 151
Report of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys 153
Report of the Research Committee on Fatigue of Metals 157
Appendix. Nomenclature for Various Ranges in Stress in Fatigue 159
Discussion 162
Report of Joint Research Committee on Effect of Temperature on the Properties of Metals 164
Appendix I. Proposed Revision of Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 34 T) 173
Appendix II. Proposed Revision of Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 34 T) 176
Appendix III. Report on Long-Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel and 0.35 per cent Carbon Steel 178
Appendix IV. Discrepancies in the Load-Carrying Abilities of Carbon Steels at 850 F.—H. W. Gillett 187
Discussion 192

NON-FERROUS METALS

Report of Committee B-1 on Copper and Copper Alloy Wires for Electrical Conductors 193
Report of Committee B-2 on Non-Ferrous Metals and Alloys 198
Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys 201
Report of Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys 203
Discussion 207

PAGE
Report of Committee B-5 on Copper and Copper Alloys, Cast and Wrought........ 208
Report of Committee B-6 on Die-Cast Metals and Alloys........................ 211
  Appendix I. Brass Die Castings—J. C. Fox.................................. 215
  Appendix II. A Study of Die Design Changes for the Improvement of the Soundness and Uniformity of Test Bars—G. L. Werley.................... 223
Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought......... 255
  Appendix I. Modulus of Elasticity of Aluminum Alloys—R. L. Templin and Sam Tour.. 258
  Appendix II. Report on Methods of Testing Oxide Coatings on Aluminum..... 261

CEMENTITIOUS, CERAMIC, CONCRETE, AND MASONRY MATERIALS

Report of Committee C-1 on Cement.. 273
  Report of Working Committee on Fineness.................................... 280
  Report of Working Committee on Plastic Mortar Tests......................... 282
  Report of Working Committee on Methods of Chemical Analysis of Portland Cement.. 284
  Report of Subcommittee on Cement Reference Laboratory...................... 288
  Appendix I. Procedure for Making Autoclave Tests of Portland Cement Used as Basis of Comparison in Current Cooperative Studies................. 290
  Appendix II. Proposed Tentative Specifications and Methods of Test for Blended Cements... 291
Report of Committee C-3 on Brick.. 300
Report of Committee C-5 on Fire Tests of Materials and Construction........... 305
Report of Committee C-8 on Refractories....................................... 307
Report of Committee C-9 on Concrete and Concrete Aggregates................... 311
  Appendix. A Study of Plastic Mortar Cubes—H. W. Leavitt................... 321
Report of Committee C-10 on Hollow Masonry Building Units...................... 333
Report of Committee C-11 on Gypsum... 335
Report of Committee C-13 on Concrete Pipe...................................... 337

MISCELLANEOUS MATERIALS: PRESERVATIVE COATINGS, ETC.

Report of Committee D-1 on Paint, Varnish, Lacquer, and Related Products........ 339
  Report of Subcommittee III on Testing of Paint Vehicles...................... 347
  Report of Subcommittee VII on Accelerated Tests for Protective Coatings...... 350
  Report of Subcommittee VIII on Methods of Analysis of Paint Materials....... 353
Report of Committee D-2 on Petroleum Products and Lubricants................... 360
  Report of Subcommittee XI on Turbine Oils.................................... 367
  Report of Subcommittee XXII on Natural Gasoline.............................. 369
  Appendix I. Proposed Method of Test for Dropping Point of Lubricating Greases 373
  Appendix II. Proposed Method of Test for Unsulfonated Residue of Plant Spray Oils... 375
  Appendix III. Method for Converting Kinematic Viscosity to Saybolt Universal Viscosity—C. E. Headington and J. C. Geniesse........................ 380
  Appendix IV. Diesel-Fuel-Oil Classification.................................. 385
Report of Sectional Committee on Petroleum Products and Lubricants............. 389
Report of Committee D-3 on Gaseous Fuels.. 391
Report of Committee D-4 on Road and Paving Materials........................... 395
Report of Committee D-5 on Coal and Coke.. 402
Report of Sectional Committee on Classification of Coals....................... 406
Report of Committee D-7 on Timber... 410

PAGE
Report of Committee D-8 on Bituminous Waterproofing and Roofing Materials...... 413
Report of Committee D-9 on Electrical Insulating Materials........................ 424
  Appendix I. Proposed Revisions in Standards for Electrical Insulating Materials.. 430
  Appendix II. Significance of Tests of Electrical Insulating Materials:
    Significance of the Dielectric Strength Test—Dean Harvey................ 436
    Significance of the Resistivity Test—H. L. Curtis........................ 439
    Significance of the Impact Test—M. P. Davis.............................. 443
Report of Committee D-11 on Rubber Products.. 444
Report of Committee D-12 on Soaps and Detergents................................... 454
  Appendix I. Proposed Methods of Chemical Analysis of Sulfonated (Sulfated) Oils.. 457
  Appendix II. Proposed Methods of Chemical Analysis of Special Detergents... 477
Report of Committee D-13 on Textile Materials...................................... 481
  Appendix. Proposed Revisions in Standards for Textile Materials............ 491
Report of Committee D-17 on Naval Stores... 501
  Report of Subcommittee IV on Acid and Saponification Number and Unsaponifiable Matter... 503
Report of Committee D-19 on Water for Industrial Uses.............................. 505

MISCELLANEOUS SUBJECTS

Report of Committee E-1 on Methods of Testing...................................... 508
  Appendix I. Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals.. 518
  Appendix II. Yield Strengths Corresponding to Small Percentages of Set....... 523
Report of Committee E-2 on Spectrographic Analysis................................. 528
  Report of Subcommittee V on Standards and Pure Materials.................... 531
  Appendix. Super-Purity Metals—Thomas A. Wright.............................. 538
Report of Committee E-3 on Chemical Analysis of Metals............................ 543
Report of Committee E-4 on Metallography... 546
Report of Committee E-9 on Research.. 549
Report of Committee E-10 on Standards.. 555

A.S.T.M. TENTATIVE STANDARDS

ISSUED OR REVISED, 1937

A. FERROUS METALS

STEEL

Boiler Steel

Tentative Specifications for:

A 201 – 37 T. Carbon-Silicon-Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels.......... 565
A 202 – 37 T. Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels.............................. 572
A 203 – 37 T. Low-Carbon-Nickel Steel Plates for Boilers and Other Pressure Vessels.. 577
A 204 – 37 T. Molybdenum-Steel Plates for Boilers and Other Pressure Vessels. 582

Steel for Welding

Tentative Specifications for:

PAGE

A 205 – 37 T. Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods) 589

Steel Tube and Pipe

Tentative Specifications for:

A 199 – 37 T. Heat-Exchanger and Condenser Tubes, Intermediate Alloy-Steel, Seamless Cold-Drawn 601

A 200 – 37 T. Still Tubes for Refinery Service, Intermediate Alloy-Steel, Seamless 608

A 158 – 37 T. Pipe for Service at Temperatures from 750 to 1100 F., Seamless Alloy-Steel 622

A 206 – 37 T. Pipe for Service at Temperatures from 750 to 1000 F., Seamless Carbon-Molybdenum Alloy-Steel 633

Steels for High-Temperature Service

Tentative Specifications for:

A 193 – 37 T. Bolting Materials, Alloy-Steel, for High-Temperature Service from 750 to 1100 F. Metal Temperatures 616

A 158 – 37 T. Pipe, Seamless Alloy-Steel, for Service at Temperatures from 750 to 1100 F. 622

A 206 – 37 T. Pipe, Seamless Carbon-Molybdenum Alloy-Steel, for Service at Temperatures from 750 to 1000 F. 633

A 199 – 37 T. Heat-Exchanger and Condenser Tubes, Seamless Cold-Drawn Intermediate Alloy-Steel 601

A 200 – 37 T. Still Tubes for Refinery Service, Seamless Intermediate Alloy-Steel .. 608

Tentative Methods of:

E 21 – 37 T. Tension Tests, Short-time High-Temperature, of Metallic Materials, Test for 1290

Zinc-Coated Iron and Steel Articles

Tentative Specifications for:

A 121 – 37 T. Barbed Wire, Zinc-Coated (Galvanized) Iron or Steel 643

A 116 – 37 T. Farm-Field and Railroad Right-of-Way Wire Fencing, Zinc-Coated (Galvanized) Iron or Steel 646

WROUGHT IRON

Tentative Specifications for:

A 189 – 37 T. Bars, Single and Double Refined Wrought-Iron 649

A 42 – 37 T. Plates, Wrought-Iron 653

A 152 – 37 T. Rivets and Rivet Rounds, Wrought-Iron 659

A 207 – 37 T. Shapes and Bars, Rolled Wrought-Iron 664

FERROUS METALS, GENERAL

Tentative Methods of:

E 21 – 37 T. Tension Tests, Short-Time High-Temperature, of Metallic Materials, Test for 1290

B. NON-FERROUS METALS

ALUMINUM AND ALUMINUM ALLOYS

Tentative Specifications for:

PAGE
B 58 – 37 T. Alloys in Ingot Form for Sand-Castings, Aluminum-Base...... 668
B 112 – 37 T. Alloys in Ingot Form for Permanent Mold Castings, Aluminum-Base.. 671
B 85 – 37 T. Die Castings, Aluminum-Base Alloy........................ 705
B 108 – 37 T. Permanent Mold Castings, Aluminum-Base Alloy............ 674
B 26 – 37 T. Sand Castings, Aluminum-Base Alloy...................... 678

MAGNESIUM AND MAGNESIUM ALLOYS

Tentative Specifications for:

B 94 – 37 T. Die Castings, Magnesium-Base Alloy....................... 708

COPPER AND COPPER ALLOYS

Copper and Copper Alloy Tubes

Tentative Specifications for:

B 111 – 37 T. Condenser Tubes and Ferrule Stock, Seamless, Copper and Copper-Alloy.. 684

Copper-Silicon Alloys

Tentative Specifications for:

B 99 – 37 T. Wire for General Purposes, Copper-Silicon Alloy............. 689

Copper Wire and Cable

Tentative Specifications for:

B 105 – 37 T. Copper Alloy Wires for Electrical Conductors, Hard-Drawn... 693
D 27 – 37 T. Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound.. 1070
D 469 – 37 T. Insulated Wire and Cable: Heat-Resisting Rubber Compound.... 1096
D 353 – 37 T. Insulated Wire and Cable: Performance Rubber Compound...... 1109
B 99 – 37 T. Wire for General Purposes, Copper-Silicon Alloy.............. 689

Zinc

Tentative Specifications for:

B 69 – 37 T. Rolled Zinc.. 698

DIE CASTINGS

Tentative Specifications for:

B 85 – 37 T. Aluminum-Base Alloy Die Castings........................ 705
B 94 – 37 T. Magnesium-Base Alloy Die Castings....................... 708

ELECTRICAL-HEATING AND ELECTRICAL-RESISTANCE ALLOYS

Tentative Methods of:

B 106 – 37 T. Flexivity of Thermoflex (Thermostatic Metals), Test for....... 711
E 21 – 37 T. Tension Tests, Short-Time High-Temperature, of Metallic Materials, Test for.. 1290

NON-FERROUS METALS, GENERAL

Tentative Method of:

E 21 – 37 T. Tension Tests, Short-Time High-Temperature, of Metallic Materials, Test for.. 1290

METHODS OF SPECTROCHEMICAL ANALYSES OF METALS

Tentative Methods of: PAGE

E 25 – 37 T. Quantitative Spectrochemical Analysis of High Grade Pig Lead[1]
E 27 – 37 T. Quantitative Spectrochemical Analysis of Zinc Alloy Die Castings for Minor Constituents and Impurities.[1]

C. CEMENTITIOUS, CERAMIC, CONCRETE AND MASONRY MATERIALS

CEMENT

Tentative Methods of:

C 114 – 37 T. Chemical Analysis of Portland Cement 717
C 109 – 37 T. Compressive Strength of Portland-Cement Mortars, Test for... 726

MASONRY BUILDING UNITS

Brick

Tentative Specifications for:

C 62 – 37 T. Building Brick (Made from Clay or Shale) 733
C 126 – 37 T. Building Units, Glazed 736
C 7 – 37 T. Paving Brick 740
C 32 – 37 T. Sewer Brick (Made from Clay or Shale) 749

Concrete Masonry Units

Tentative Specifications for:

C 129 – 37 T. Concrete Units for Non-Load-Bearing Masonry, Specifications and Tests for 752

Fire Tests

Tentative Specifications for:

C 132 – 37 T. Fire-Retardant Properties of Wood for Scaffolding and Shoring.. 756

Refractories, Fire Brick

Tentative Methods of:

C 18 – 37 T. Chemical Analysis of Refractory Materials 760
C 133 – 37 T. Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes, Tests for 785

Tentative Definitions of:

C 71 – 37 T. Refractories, Terms Relating to 790

CONCRETE AND CONCRETE AGGREGATES

See also **Road Materials**

Tentative Specifications for:

C 33 – 37 T. Aggregates, Concrete 792
C 130 – 37 T. Lightweight Aggregate for Concrete 798
D 448 – 37 T. Standard Sizes of Coarse Aggregates for Highway Construction.... 918
C 129 – 37 T. Concrete Units for Non-Load-Bearing Masonry, Specifications and Tests for 752

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[1] These methods of spectrochemical analyses have been published as 1937 Supplements to the compilation of "A.S.T.M. Methods of Chemical Analyses of Metals, 1936," a separate publication containing all A.S.T.M. methods of chemical analyses of metals.

Tentative Methods of: PAGE

C 131 – 37 T. Abrasion of Coarse Aggregate by Use of the Los Angeles Machine, Test for........ 801
D 289 – 37 T. Abrasion of Gravel by Use of the Deval Machine, Test for........ 921
C 88 – 37 T. Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate, Test for........ 804

D. MISCELLANEOUS MATERIALS

COAL AND COKE

Tentative Methods of:

D 408 – 37 T. Grindability of Coal by the Ball-Mill Method, Test for........ 810
D 409 – 37 T. Grindability of Coal by the Hardgrove-Machine Method, Test for 815
D 440 – 37 T. Drop Shatter Test for Coal........ 820
D 441 – 37 T. Tumbler Test for Coal........ 827

TIMBER AND TIMBER PRESERVATIVES

Tentative Method of:

D 453 – 37 T. Tar Acids in Creosote and Creosote-Coal Tar Solutions, Test for........ 832

Naval Stores

Tentative Methods of:

D 465 – 37 T. Acid Number of Rosin, Test for........ 835
D 464 – 37 T. Saponification Number of Rosin, Test for........ 836

PAINT, VARNISH, LACQUER AND RELATED PRODUCTS

Pigments

Tentative Specifications for:

D 82 – 37 T. Basic Sulfate White Lead........ 838
D 442 – 37 T. Titanium-Magnesium Pigment........ 840
D 443 – 37 T. Zinc Sulfide Magnesium Pigment........ 842

Tentative Methods of:

D 406 – 37 T. Hiding Power of White Pigments, Relative Dry, Test for........ 844
D 444 – 37 T. Routine Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow) 849
D 283 – 37 T. Routine Analysis of Dry Cuprous Oxide........ 855

Oils and Thinners

Tentative Specifications for:

D 124 – 37 T. Soybean Oil, Raw........ 860

Shellac and Varnish

Tentative Methods of:

D 411 – 37 T. Shellac Used for Electrical Insulation, Testing of........ 1041

Miscellaneous Paint and Pigment Tests

Tentative Methods of:

D 344 – 37 T. Hiding Power of Paints, Relative Dry, Test for........ 866
D 406 – 37 T. Hiding Power of White Pigments, Relative Dry, Test for........ 844
D 307 – 37 T. Spectral Apparent Reflectivity of Paints, Test for........ 869

PETROLEUM PRODUCTS AND LUBRICANTS

See also **Road Materials** and **Waterproofing and Roofing Materials**

Tentative Specifications for:

PAGE

D 439 – 37 T. Gasoline 872

Tentative Methods of:

D 447 – 37 T. Distillation of Plant Spray Oils, Test for 875

†D 357 – 37 T. Knock Characteristics of Motor Fuels, Test for 879

D 323 – 37 T. Vapor Pressure of Petroleum Products (Reid Method), Test for 888

D 445 – 37 T. Kinematic Viscosity, Test for 900

D 446 – 37 T. Kinematic Viscosity to Saybolt Universal Viscosity, Conversion of 911

†D 341 – 37 T. Viscosity-Temperature Charts for Liquid Petroleum Products.. 914

ROAD MATERIALS

See also **Soils** and **Concrete and Concrete Aggregates**

Tentative Specifications for:

D 448 – 37 T. Coarse Aggregates for Highway Construction, Standard Sizes of 918

C 33 – 37 T. Concrete Aggregates 792

C 130 – 37 T. Lightweight Aggregate for Concrete 798

Tentative Methods of:

D 289 – 37 T. Abrasion of Gravel by Use of the Deval Machine, Test for..... 921

C 131 – 37 T. Abrasion of Coarse Aggregate by Use of the Los Angeles Machine, Test for 801

WATERPROOFING AND ROOFING MATERIALS

Tentative Specifications for:

D 371 – 37 T. Asphalt Cap Sheet Surfaced with Coarse Mineral Granules.... 924

D 449 – 37 T. Asphalt for Damp-proofing and Waterproofing 927

D 312 – 37 T. Asphalt for Use in Constructing Built-Up Roof Coverings..... 930

D 249 – 37 T. Asphalt Roofing Surfaced with Coarse Mineral Granules....... 933

D 248 – 37 T. Asphalt Roofing Surfaced with Fine Mineral Granules......... 937

D 224 – 37 T. Asphalt Roofing Surfaced with Powdered Talc or Mica 941

D 225 – 37 T. Asphalt Shingles Surfaced with Coarse Mineral Granules 945

D 450 – 37 T. Coal-Tar Pitch for Roofing, Damp-proofing and Waterproofing 948

D 43 – 37 T. Creosote for Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing 951

Tentative Methods of:

D 313 – 37 T. Coarse Particles in Mixtures of Asphalt and Mineral Matter, Test for 953

D 466 – 37 T. Films Deposited from Bituminous Emulsions, Testing of...... 955

D 228 – 37 T. Roll Roofing, Cap Sheets and Shingles, Asphalt, Testing of.... 957

D 451 – 37 T. Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles, Test for 965

D 452 – 37 T. Sieve Analysis of Non-Granular Mineral Surfacing for Asphalt Roofing and Shingles, Test for 969

---

† Approved as "American Tentative Standard" by the American Standards Association.

ELECTRICAL INSULATING MATERIALS

Tentative Specifications for: PAGE

D 120 – 37 T. Gloves, Rubber, for Electrical Workers on Apparatus or Circuits not Exceeding 3000 Volts to Ground........................ 1062
D 27 – 37 T. Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound.. 1070
D 469 – 37 T. Insulated Wire and Cable: Heat-Resisting Rubber Compound.... 1096
D 353 – 37 T. Insulated Wire and Cable: Performance Rubber Compound...... 1109
D 467 – 37 T. Phenolic Laminated Sheet for Radio Applications............ 973
D 373 – 37 T. Tape, Black Bias-Cut Varnished Cloth, Used for Electrical Insulation... 979
D 372 – 37 T. Tubing, Flexible Varnished, Used in Electrical Insulation...... 985

Tentative Methods of:

D 176 – 37 T. Compounds, Solid Filling and Treating, Used for Electrical Insulation, Testing of.. 988
D 468 – 37 T. Glass Insulators, Lime, Pin-Type, Testing of................ 1008
D 257 – 37 T. Insulation Resistance of Electrical Insulating Materials, Tests for 1016
D 229 – 37 T. Sheet and Plate Materials Used in Electrical Insulation, Testing of... 1027
D 411 – 37 T. Shellac Used for Electrical Insulation, Testing of............ 1041
D 295 – 37 T. Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation, Testing of.................................. 1047
D 350 – 37 T. Varnished Tubing, Flexible, Used for Electrical Insulation, Testing of... 1056

RUBBER PRODUCTS

Tentative Specifications for:

D 120 – 37 T. Gloves, Rubber, for Electrical Workers on Apparatus or Circuits Not Exceeding 3000 Volts to Ground........................ 1062
D 27 – 37 T. Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound.. 1070
D 469 – 37 T. Insulated Wire and Cable: Heat-Resisting Rubber Compound.. 1096
D 353 – 37 T. Insulated Wire and Cable: Performance Rubber Compound... 1109

Tentative Methods of:

D 454 – 37 T. Air Pressure Heat Test of Vulcanized Rubber................ 1122
D 395 – 37 T. Compression Set of Vulcanized Rubber, Tests for............ 1127
D 394 – 37 T. Abrasion Resistance of Rubber Compounds, Tests for......... 1134
D 471 – 37 T. Changes in Properties of Rubber and Rubber-Like Materials in Liquids, Tests for... 1148
D 380 – 37 T. Hose, Rubber, Testing of.................................. 1153
D 470 – 37 T. Insulated Wire and Cable, Rubber, Testing of.............. 1166

TEXTILE MATERIALS

Tentative Methods of:

D 276 – 37 T. Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles, Tests for.............................. 1174
D 414 – 37 T. Cotton Fibers, Tests for Properties of (General).............. 1189
D 258 – 37 T. Rayon, Tests and Tolerances for........................... 1202
D 472 – 37 T. Wool Tops, Specifications and Tests for Fineness of........... 1218
D 461 – 37 T. Wool Felt, Tests for....................................... 1226

Tentative Methods of: PAGE

D 462 – 37 T. Wool (Certain) and Part Wool Fabrics, Tests and Tolerances for 1231
D 418 – 37 T. Pile Floor Covering, Testing of 1235
D 463 – 37 T. Volumetric Determination of Small Amounts of Copper in Textiles, Test for 1240

Tentative Definitions of:

D 123 – 37 T. Textile Materials, Terms Relating to 1243

SOAPS AND DETERGENTS

Tentative Specifications for:

D 456 – 37 T. Caustic Soda 1260
D 457 – 37 T. Modified Soda (Sesquicarbonate Type) 1261
D 458 – 37 T. Soda Ash 1262
D 455 – 37 T. Toilet Soap, Milled 1264

Tentative Methods of:

D 460 – 37 T. Sampling and Chemical Analysis of Soaps and Soap Products 1266

Tentative Definitions of:

D 459 – 37 T. Soaps and Detergents, Terms Relating to 1289

E. GENERAL TESTING METHODS

Tentative Methods of:

E 21 – 37 T. Tension Tests, Short-Time High-Temperature, of Metallic Materials, Test for 1290

Tentative Definitions of:

E 24 – 37 T. Rheological Properties of Matter, Terms Relating to 1300

METHODS OF SPECTROCHEMICAL ANALYSES OF METALS

Tentative Methods of:

E 25 – 37 T. Quantitative Spectrochemical Analysis of High Grade Pig Lead.[1]
E 27 – 37 T. Quantitative Spectrochemical Analysis of Zinc Alloy Die Castings for Minor Constituents and Impurities.[1]

TENTATIVE REVISIONS OF A.S.T.M. STANDARDS

A. FERROUS METALS

Revisions of Standard Specifications for:

A 30 – 33. Boiler and Firebox Steel for Locomotives 1303
A 149 – 36. High Tensile Strength Carbon-Steel Plates for Pressure Vessels (Plates 2 in. and Under in Thickness) 1303
A 150 – 36. High Tensile Strength Carbon-Steel Plates for Fusion-Welded Pressure Vessels (Plates over 2 in. to 4 in., Inclusive, in Thickness) 1304
A 141 – 36. Structural Rivet Steel 1305
A 96 – 33. Alloy-Steel Bolting Material for High-Temperature Service 1306

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Revisions of Standard Specifications for: PAGE

A 56 – 30. Iron and Steel Chain 1307
A 72 – 33. Welded Wrought-Iron Pipe 1308
A 73 – 36. Wrought-Iron Rolled or Forged Blooms and Forgings 1308
A 84 – 36. Staybolt Wrought Iron, Solid 1308
A 86 – 36. Staybolt Wrought Iron, Hollow Rolled 1310
A 162 – 36. Uncoated Wrought-Iron Sheets 1310
A 163 – 36. Zinc-Coated (Galvanized) Wrought-Iron Sheets 1310
A 116 – 30. Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing 1310
A 121 – 30. Zinc-Coated (Galvanized) Iron or Steel Barbed Wire 1310

B. NON-FERROUS METALS

Revisions of Standard Specifications for:

B 7 – 27. Manganese-Bronze Ingots for Sand Castings 1310
B 54 – 27. Manganese-Bronze Sand Castings 1311
B 69 – 29. Rolled Zinc 1311

C. CEMENTITIOUS, CERAMIC, CONCRETE AND MASONRY MATERIALS

Revisions of Standard Specifications for:

C 5 – 26. Quicklime for Structural Purposes 1311
C 6 – 31. Hydrated Lime for Structural Purposes 1311
C 14 – 35. Concrete Sewer Pipe 1311
C 62 – 30. Building Brick (Made from Clay or Shale) 1312

Revisions of Standard Specifications for:

C 82 – 34. Curing Portland-Cement Concrete Slabs with Calcium-Chloride Admixture 1312
C 83 – 36. Curing Portland-Cement Concrete Slabs by Surface Application of Calcium Chloride 1313
C 94 – 35. Ready Mixed Concrete 1313

Revision of Standard Method of:

C 31 – 33. Making and Storing Compression Test Specimens of Concrete in the Field 1315

Revisions of Standard Definitions of:

C 71 – 36. Terms Relating to Refractories 1315
C 11 – 28. Terms Relating to the Gypsum Industry 1316
C 58 – 28. The Term Sand 1316

D. MISCELLANEOUS MATERIALS

Revisions of Standard Specifications for:

D 82 – 24. Basic Sulfate White Lead 1316
D 79 – 24. Zinc Oxide 1316
D 386 – 36. Zinc Sulfide 1316
D 208 – 26. Lithopone 1316
D 125 – 23. Perilla Oil, Raw or Refined 1317
D 12 – 33. Raw Tung Oil 1317

Revisions of Standard Methods of:

D 29 – 33. Sampling and Testing Shellac 1320
D 154 – 28. Testing of Oleo-Resinous Varnishes 1320

Revisions of Standard Methods of:

PAGE
D 283 – 36. Routine Analysis of Dry Cuprous Oxide........................ 1320
D 88 – 36. Test for Viscosity by Means of the Saybolt Viscosimeter......... 1320
D 61 – 24. Test for Softening Point of Tar Products (Cube-in-Water Method) 1321

Revisions of Standard Specifications for:

D 65 – 23. Broken Slag for Waterbound Base and Wearing Course.......... 1321
D 66 – 23. Shovel-Run or Crusher-Run Broken Slag for Waterbound Base... 1321
D 59 – 26. Block for Granite Block Pavements.......................... 1322
D 131 – 23. Block for Recut Granite Block Pavements...................... 1322
D 132 – 23. Block for Durax Granite Pavements.......................... 1322

Revisions of Standard Methods of:

D 39 – 36. Testing Woven Textile Fabrics................................ 1322
D 179 – 33. Testing and Tolerances for Tire Cord, Woven and on Cones...... 1323
D 116 – 34. Testing Electrical Porcelain................................ 1324
D 117 – 36. Testing Electrical Insulating Oils.......................... 1324
D 257 – 33. Test for Resistivity of Insulating Materials..................... 1324
D 21 – 16. Sampling Coal.. 1325
D 271 – 37. Laboratory Sampling and Analysis of Coal and Coke............ 1326

Revisions of Standard Definitions of:

D 121 – 30. Terms Relating to Coal and Coke........................... 1327
D 324 – 33. Terms Relating to Timber Preservatives...................... 1327

Officers and Members of Executive Committee............................ 1328
Subject Index.. 1329
Author Index... 1361

American Society for Testing Materials

ORGANIZED IN 1898
INCORPORATED IN 1902

PROCEEDINGS, PART I

The Society is not responsible, as a body, for the statements and opinions advanced in this publication.

The following Summary records the action taken by the Society at the Annual Meeting on the Recommendations Affecting Standards and Tentative Standards appearing in the Annual Reports of the Standing Committees. In the individual committee reports attention is called through an editorial footnote to any modifications of the recommendations or where the recommendations of the committee were not approved. In all other instances it is to be understood that the recommendations were approved as submitted by the committee.

SUMMARY OF THE PROCEEDINGS OF THE FORTIETH ANNUAL MEETING

New York City, June 28–July 2, 1937

The Fortieth Annual Meeting of the American Society for Testing Materials was held at The Waldorf-Astoria, New York City, June 28–July 2, 1937. The following is an analysis of the registered attendance at the meeting: Members in attendance or represented, 960; committee members, 186; guests, 377; total 1523; ladies, 179. The corresponding statistics for the Thirty-ninth Annual Meeting are as follows: Members in attendance or represented, 757; committee members, 143; guests, 231; total 1131; ladies, 256.

The Fourth Exhibit of Testing Apparatus and Related Equipment sponsored by the Society was held throughout the five days of the annual meeting. The Exhibit was the most extensive of any yet held and was of outstanding interest to the members and large number of visitors. In the 57 booths there was a wide range of testing and scientific instruments, laboratory apparatus and supplies and related products. Many of the leading companies in the industry sponsored displays, 42 participating. The companies featured new equipment and products, some of which were displayed for the first time.

Features of the Exhibit were the displays sponsored by six Society committees as follows: A-5 on Corrosion of Iron and Steel (in cooperation with Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys and other A.S.T.M. committees); B-7 on Light Metals and Alloys, Cast and Wrought; E-4 on Metallography; D-9 on Electrical Insulating Materials; D-13 on Textile Materials and the Joint A.S.M.E.-A.S.T.M. Research Committee on Effect of Temperature on the Properties of Metals. Each of these committees emphasized various phases of its work, the groups as a whole giving a conception of the wide extent of A.S.T.M. work in standardization and research. There were four displays sponsored by industrial and institutional research laboratories featuring apparatus and equipment used for special research and testing work.

FIRST SESSION—MONDAY, JUNE 28, 3.30 P. M.

Formal Opening Session

President Arno C. Fieldner in the chair.

President Fieldner in opening the Fortieth Annual Meeting called attention to the fact that this was the first annual meeting that the Society has held in New York City since 1912, at the time of the Sixth Congress of the International Association for Testing Materials. President Fieldner stated that from the attendance records, the number of scheduled meetings and technical sessions, the fine exhibit of testing apparatus, and other scheduled activities, this meeting promised to be the most successful that the Society has yet held. He said that the Society had been especially active during the year, as evidenced by the large number of meetings of standing committees that had been held which resulted in annual reports from 50 committees to be submitted at this annual meeting, including a large number of recommendations on new and revised standards. He referred particularly to the activities of the eight district committees of the Society, under whose auspices several meetings had been held during the year. President Fieldner in referring to the very satisfactory increase in membership which had taken place during the year, called attention to the fact that the Society was extending its activities into new materials fields which should result in the Society rendering an increasing service to its membership and to industry.

President Fieldner then presented the Honorary Chairman of the New York Committee on Arrangements, Past-President H. S. Vassar, who extended a cordial welcome to the Society on behalf of the members in the New York District. He stated that the New York Committee was indeed pleased to act as host to the Society membership on this occasion of its Fortieth Annual Meeting and expressed the hope that the arrangements that had been made would contribute to a most successful meeting.

Mr. Vassar then introduced M. F. Skinker, chairman of the New York Committee on Arrangements, who outlined briefly the plans and arrangements that had been made for the entertainment of the members and guests and particularly of the ladies during the meeting. He said that no effort had been spared by the committee to make this a grand occasion.

The President then introduced P. H. Bates, chairman of Committee E-9 on Research, who presented the report of the committee.

The President then presented Cloyd M. Chapman, chairman of Committee E-10 on Standards, who presented the report of the committee.

President Fieldner then introduced the Secretary-Treasurer, C. L. Warwick, who presented the report of the Executive Committee. This report on general Society activities covered membership, publications, finances, and administrative matters relating to committee activities and inter-society relations. The proposed revisions in the By-laws of the Society were approved for reference to letter ballot of the Society for adoption.

Past-President Hermann von Schrenk then assumed the chair.

The chairman presented the President, Arno C. Fieldner, who delivered the annual Presidential Address on "Fuels of Today and Tomorrow," in which he traced the use of the several fuels for the past 30 yr. In reviewing the national reserves of fuels he pointed out that although there is sufficient coal for hundreds and possibly thousands of years, the natural gas and oil obtainable by present methods may be exhausted in 100 yr and a shortage of our domestic supply may begin within 10 or 20 yr. In presenting an appraisal of present trends and future probabilities of the fuels of tomorrow, President Fieldner predicted that coal will continue to be the principal fuel used for the generation of major industrial and public utility power but that it will be burned more efficiently as the result of technologic improvements. He sees no substitute for metallurgical coke but anticipated the wider use of the by-product gases industrially. In domestic applications he indicated that the stoker-fired furnaces will tend to halt the inroads being made by oil in house heating.

In discussing the very interesting question of the motor fuel supply, he indicated that the use of scientific methods in extracting oil from sands, and catalytic polymerization and hydrogenation eventually may furnish the means for complete conversion of volatile liquids and heavy petroleum to gasoline. None of these new fuels, however, can hope to be as cheap as our present petroleum fuels. In conclusion, President Fieldner pointed out that although gas, oil and coal have energized an industrial civilization on a magnificent scale, we are awakening to the need of conserving these resources wisely so that the generations of tomorrow will have better fuels than we of today. Future scientists may unlock undreamed of powers, but engineers should plan on the basis of the resources known today.

For the Award of Honorary Membership, the chairman requested N. L. Mochel to escort John A. Capp to the platform, W. R. Webster to escort Guilliam H. Clamer, and C. S. Reeve to escort Gustave W. Thompson. Each in turn was presented to the President who made the award of the Honorary Membership Certificate on behalf of the Executive Committee which had unanimously elected them as Honorary Members in recognition of the eminent position each had attained in the field of metals testing and in acknowledgment of the many years of service given in the cause of engineering standardization.

The chairman then recognized H. F. Moore, chairman of the Committee of Tellers, who reported the results of the letter ballot on election of officers. Of the 1203 ballots cast, the results were as follows:

For President, to serve for one year: A. E. White, 1195 votes.

For Vice-President, to serve for two years: H. H. Morgan, 1192 votes.

For Members of Executive Committee, to serve for two years:

P. H. Bates, 1181 votes.
H. F. Clemmer, 1176 votes.
G. E. F. Lundell, 1178 votes.
H. C. Mougey, 1183 votes.
R. L. Templin, 1176 votes.

In accordance with the ballot the chair declared the officers listed above elected.

The chairman then introduced the newly elected members of the Executive Committee.

At the request of the chairman, Past-President H. S. Vassar and Allen Rogers escorted the Vice-President-Elect, H. H. Morgan, to the chair, who in expressing his appreciation of the honor of his election stated that he realized its acceptance carries with it certain responsibilities which he was prepared to accept.

The chairman then requested Past-Presidents F. O. Clements and H. F. Moore to escort to the chair the President-Elect, A. E. White.

The meeting then adjourned till 8 p. m.

MONDAY, JUNE 28, 8 P. M.

Round-Table Discussion on Precision and Accuracy

Messrs. H. F. Dodge and G. E. Hopkins, co-chairmen.

A Round-Table Discussion on Precision and Accuracy was held on Monday evening, June 28, at 8 p. m. This informal round-table meeting was arranged under the joint sponsorship of Technical Committee IX on Interpretation and Presentation of Data (H. F. Dodge, chairman), and Technical Committee XI on Designation and Interpretation of Numerical Requirements (J. A. Gann, chairman), of Committee E-1 on Methods of Testing.

In introducing this first general discussion before the Society on this subject, H. F. Dodge and G. E. Hopkins, co-chairmen of the round-table discussion, reviewed briefly the reasons for arranging this program for the purpose of clarifying the understanding of engineers with respect to the significance and use of the terms "precision," "accuracy," "sensitivity," and "reproducibility of results." The several phases of the subject considered at the meeting were introduced by the following three formal discussions:

"The Fundamental Aspects of the Subject," by W. A. Shewhart.

"The Field of Mechanical Testing," by H. F. Moore.

"The Field of Chemical Analysis," by G. E. F. Lundell.

A prepared discussion of each of the above formal contributions was then presented, respectively, by R. F. Passano, H. J. Ball and H. V. Churchill, following which the subject of the meeting was informally discussed by those in attendance. At the close of the meeting a resolution for submission to Committee E-1 on Methods of Testing was adopted recommending "that when a standing committee records or specifies a numerical value for precision in a standard, it make clear what is meant in terms of operations or procedures to be followed for purposes of verification."

The meeting then adjourned till the following morning.

SECOND SESSION—TUESDAY, JUNE 29, 9.30 A. M.

Symposium on Consistency: Critical Discussion on Present-Day Practices in Consistency Measurement

(Held Simultaneously with Third Session)

Past-President W. H. Fulweiler in the chair.

Mr. Fulweiler in introducing the Symposium on Consistency, summarized briefly the reasons for arranging for this series of papers to consider the advances in recent years made in consistency measurement, including a discussion of the theoretical background and the existing nomenclature. He called attention to the fact that the nine papers comprising the symposium covered a representative cross-section of engineering materials concerning which the establishment of authoritative nomenclature and fundamental principles underlying the measurement of consistency, plasticity, viscosity and related properties is important. Mr. Fulweiler stated that it was the purpose of the symposium to present a critical review of consistency measurement as applied to these materials, and to consider the possibilities of standardization and the use and advantages to be derived from the use of fundamental units of measurement.

Mr. Fulweiler then introduced the first paper entitled "Recent Progress in Consistency Measurement," by E. C. Bingham, which was presented by the author and discussed.

The co-chairmen, Messrs. E. C. Bingham, H. A. Gardner, C. S. Reeve, L. C. Beard, Jr., Arthur W. Carpenter and T. Smith Taylor, then presided in turn during the presentation of the remaining papers, as follows, which were presented by the authors and discussed:

"Definition of Consistency and Theoretical Considerations," by M. Mooney.

"Consistency Measurements in the Paint Industry," by D. L. Gamble.

"The Flow Properties of Asphalts Measured in Absolute Units," by R. N. Traxler.

"Consistency Measurements in the Coal-Tar Industry," by E. O. Rhodes, E. W. Volkmann and C. T. Barker (presented by Mr. Volkmann).

"Viscosity Measurement of Petroleum Products and Lubricants," by J. C. Geniesse.

"Consistency Measurement of Rubber and Rubber Compounds," by J. H. Dillon and L. V. Cooper (presented by Mr. Dillon).

"Measurements of Flow Characteristics of Thermosetting Resins," by H. L. Bender, H. F. Wakefield and H. E. Riley (presented by Mr. Riley).

"Cold Flow of Insulating Materials," by Robert Burns and Irving L. Hopkins (presented by Mr. Burns).

The meeting then adjourned till 1.30 p. m.

THIRD SESSION—TUESDAY, JUNE 29, 9.30 A. M.

Symposium on Significance of Tests of Coal

(Held Simultaneously with Second Session)

President A. C. Fieldner and Mr. J. B. Morrow, co-chairmen.

President Fieldner in opening the Symposium on Significance of Tests of Coal stated that it had been arranged for the purpose of providing the engineering profession with information on the applicability of the tests for coal now in general use in this country and the significance of the results obtained. He then introduced the members of the special committee responsible for the arrangements for the symposium: A. W. Gauger, chairman; G. B. Gould, and J. B. Morrow. The following three papers, comprising the first half of the symposium, were then presented by their authors and discussed:

"Pulverizer Performance as Affected by Grindability and Other Factors," by Martin Frisch and A. C. Foster (presented by Mr. Foster).

"The Significance of Ash-Softening Temperature and Ash Composition in the Utilization of Coal," by A. W. Gauger.

"Interpretation of Laboratory Coal Tests—Proximate Analysis and Calorific Value," by G. B. Gould.

The meeting then adjourned till 2 p. m., the symposium being continued in the Fourth Session.

FOURTH SESSION—TUESDAY, JUNE 29, 2 P. M.

Coal, Coke, Gaseous Fuels

(Held Simultaneously with Fifth and Sixth Sessions)

Mr. A. W. Gauger in the chair.

The report of Committee D-3 on Gaseous Fuels was presented by the chairman, A. C. Fieldner.

The report of Committee D-5 on Coal and Coke was presented by the chairman, A. C. Fieldner. The proposed Tentative Methods of Drop Shatter Test for Coal, and of Tumbler Test for Coal were accepted for publication as tentative. The proposed revisions of the Tentative Methods of Test for Grindability of Coal by the Ball-Mill Method (D 408 - 35 T) and Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 - 35 T) were accepted, the methods as revised being continued as tentative. The proposed revisions of the Standard Methods of Sampling Coal (D 21 - 16) and Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 33) were accepted for publication as tentative. The tentative revision of the Standard Methods D 271 was approved for reference to letter ballot of the Society for adoption as standard, this recommendation requiring a nine-tenths vote.

The report of Sectional Committee M20 on Classification of Coals was presented by the chairman, A. C. Fieldner. The Tentative Specifications for Classification of Coals by Grade (D 389 - 34 T) and for Classification of Coals by Rank (D 388 - 36 T), as revised, were approved for reference to letter ballot of the Society for adoption as standard.

Mr. G. B. Gould then assumed the chair.

The Symposium on Significance of Tests of Coal, which had been opened in the Third Session, was continued in this session with the following three papers which were presented by their authors and discussed:

"The Significance to the Consumer of Sulfur in Coal," by Henry Kreisinger.

"Laboratory Tests Relating to Caking, Plastic, Gas- and Coke-Making Properties of Bituminous Coals," by O. O. Malleis.

"Significance of Friability and Size Stability Tests on Coal," by R. E. Gilmore and J. H. H. Nicolls (presented by Mr. Gilmore).

The meeting then adjourned till the following morning.

FIFTH SESSION—TUESDAY, JUNE 29, 1.30 P. M.

Paint and Related Products

(Held Simultaneously with Fourth and Sixth Sessions)

Mr. R. L. Hallett in the chair.

The report of Committee D-1 on Paint, Varnish, Lacquer, and Related Products, was presented by the secretary, M. Rea Paul. The proposed Tentative Specifications for Zinc Sulfide Magnesium Pigment, for Titanium-Magnesium Pigment, and Methods of Routine Analysis of Zinc Yellow Pigment (Zinc-Chromate Yellow) were accepted for publication as tentative. The proposed Tentative Method of Test for Spectral Apparent Reflectivity of Paints was accepted for publication as tentative to replace the Standard Method of Analysis for the Color Characteristics of Paints in Terms of Fundamental Physical Units (D 307 - 30) which was accordingly withdrawn.

The revisions proposed for immediate adoption in the Standard Methods of Test for Coarse Particles in Dry Pigments and Coarse Particles and Skins in Mixtures of Pigments and Vehicles (D 185 - 29), of Routine Analysis of Dry Red Lead (D 49 - 35) and of Routine Analysis of Titanium Pigments (D 186 - 33) were unanimously approved for reference to letter ballot of the Society for adoption as standard, this recommendation requiring a nine-tenths vote.

The proposed revisions of the Standard Specifications for Raw Tung Oil (D 12 - 33), for Zinx Oxide (D 79 - 24), for Basic Sulfate White Lead (D 82 - 24) in the form of completely revised specifications, for Perilla Oil, Raw or Refined (D 125 - 23), for

Zinc Sulfide (D 386 - 36), and Methods of Routine Analysis of Dry Cuprous Oxide (D 283 - 36) in the form of completely revised methods, were accepted for publication as tentative.

The proposed revisions of the Tentative Specifications for Raw Soybean Oil (D 124 - 33 T), Methods of Test for Comparative Hiding Power of Paints (D 344 - 32 T) and Test for Comparative Hiding Power of White Pigments (D 406 - 35 T) were accepted, the specifications and methods as revised being continued as tentative. The tentative revision of the Standard Methods of Routine Analysis of White Linseed Oil Paints (D 215 - 35) was unanimously approved for reference to letter ballot of the Society for adoption as standard, this recommendation requiring a nine-tenths vote.

The recommendation presented on the floor at the annual meeting for the adoption as standard of the Tentative Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (D 333 - 34 T) revised as indicated below, was unanimously approved for reference to letter ballot of the Society, subject to favorable letter ballot of Committee D-1:[1]

Section 6.—Delete the description of the bending test, which reads as follows, renumbering the remaining sections of the methods accordingly:

> 6. The composition of the metal test panel for the bending test, the gage thereof and the manner of application of lacquer coating applied thereto, shall be mutually agreed upon by the buyer and seller.
>
> The test shall be made by bending the panel at a uniform rate through an angle of more than 140 deg. over a mandrel of mutually agreed upon radius.

A report on house paint exposure tests for gloss retention and chalking resistance, submitted by Subcommittee VII on Accelerated Tests for Protective Coatings, presented on the floor at the annual meeting, which had been accepted by the committee for inclusion in its annual report but had not been preprinted, was accepted, subject to approval by Committee E-6 on Papers and Publications.

It was announced that the committee had completed the preparation of Proposed Specifications for c. p. Zinc Yellow (Zinc Chromate), for Para Red Toner, for Reduced Para Red, and Method of Test for Reactivity of Paint Liquids; also proposed revisions in the Standard Specifications for Normal Butyl Acetate (88 to 92 per cent Grade) (D 303 - 33), for Butyl Propionate (90 to 93 per cent Grade) (D 320 - 33) and for Ethyl Lactate (Synthetic) (D 321 - 33) which, if approved by letter ballot of the committee, would be submitted to the Society for publication as tentative through Committee E-10 on Standards subsequent to the annual meeting.

Mr. H. A. Nelson then assumed the chair.

Mr. Nelson in introducing the Symposium on Correlation Between Accelerated Laboratory Tests and Service Tests on Protective and Decorative Coatings stated that it had been arranged for the purpose of promoting the exchange of information and experience on this important subject.

Mr. Nelson then presented W. R. Fuller who opened the discussion on Topic I: Finishes for Indoor Service. Mr. Fuller introduced in turn the following who participated in the discussion of this subject:

C. C. Hipkins, Finish Engineer, Bell Telephone Laboratories.

Leo Roon, President, Roxalin Flexible Lacquer Co., Inc. (Discussion presented by Mr. Kirk, Secretary of the Company in absence of Mr. Roon.)

J. B. Bullitt, Jr., Chemist, Philadelphia Laboratory, E. I. du Pont de Nemours and Co., Inc.

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[1] This recommendation was submitted to letter ballot of Committee D-1 which consists of 214 members; 79 members returned their ballots, of whom 43 voted affirmatively and 1 negatively.

William H. Lutz, Technical Director, Pratt & Lambert, Inc.
W. C. Norris, Selden Division, American Cyanamid and Chemical Corp., who summarized his paper on "Equipment for Testing the Resistance to Cold Checking of Lacquers and Other Surface Coatings."
R. J. Moore, Development Manager, Varnish Resin Division, Bakelite Corp.
E. F. Hickson, Chemist, National Bureau of Standards.

Mr. Nelson then presented H. A. Gardner who opened the discussion on Topic II: Paints for Exterior Service on Wood. Mr. Gardner called upon each of the following:

F. L. Browne, Senior Chemist, Forest Products Laboratory.
C. D. Holley, Director of Paint Research, Sherwin-Williams Co.
M. J. Dorcas, Lamp Department Manager, National Carbon Co.
A. C. Eide, Sales Engineer, American Zinc Sales Co.
H. A. Depew, Research Chemist, American Zinc Sales Co.
G. B. Girault, Paint Technologist, Robert W. Hunt Co.
C. Y. Hopkins, Division of Chemistry, National Research Council, Canada.
E. W McMullen, Director of Research, Eagle-Picher Lead Co.
C. H Rose, Chemist, National Lead Co.
S. Werthan, Chief of Paint Section, Research Division, The New Jersey Zinc Co.

Mr. Nelson then presented C. F. Rassweiler who opened the discussion on Topic III: Paints for Iron and Steel. Mr. Rassweiler then called upon each of the following:

G. B. Girault, Paint Technologist, Robert W. Hunt Co.
S. G. Saunders, Chrysler Corp.
V. M. Darsey, Technical Director, Parker Rust-Proof Co.
E. H. Barlow, Chief Engineer, The Standard Oil Development Co. (Discussion presented by G. W. Oxley in absence of Mr. Barlow.)
C. E. Wilson, Technical Director, James B. Sipe and Co.
J. C. Moore, Superintendent, Paint Dept., Sinclair Refining Co.

The meeting then adjourned till the following morning.

SIXTH SESSION—TUESDAY, JUNE 29, 2 P. M.

Bituminous Materials

(Held Simultaneously with Fourth and Fifth Sessions)

Mr. J. S. Miller in the chair.

The report of Committee D-8 on Bituminous Waterproofing and Roofing Materials was presented by the chairman, J. M. Weiss.

The proposed Tentative Methods of Test for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles, and for Sieve Analysis of Non-Granular Mineral Surfacing for Asphalt Roofing and Shingles were accepted for publication as tentative. The proposed Tentative Specifications for Asphalt for Damp-proofing and Waterproofing were accepted for publication as tentative to replace the existing Tentative Specifications D 40 - 36 T and D 144 - 36 T; also the proposed Tentative Specifications for Coal-Tar Pitch for Roofing, Damp-proofing and Waterproofing were accepted for publication as tentative to replace the existing Tentative Specifications D 42 - 36 T, D 145 - 36 T, D 200 - 36 T, D 201 - 36 T, D 251 - 36 T and D 252 - 36 T.

The revision proposed for immediate adoption in the Standard Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (D 146 - 27) were unanimously approved for reference to letter ballot of the Society for adoption as standard, this recommendation requiring a nine-tenths vote.

The proposed revisions of the Tentative Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 - 35 T), for Asphalt Shingles Surfaced with Coarse Mineral Granules (D 225 - 35 T), for Asphalt Roofing Surfaced with Fine Mineral Granules (D 248 - 35 T), for Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 - 35 T), for Asphalt for Use in Constructing Built-Up Roof Coverings (D 312 - 25 T), for Asphalt Cap Sheet Surfaced with Mineral Granules (D 371 - 33 T), for Creosote Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing Below and Above Ground Level (D 43 - 36 T), and Methods of Testing Asphalt Roll-Roofing Surfaced with Fine Talc, Granular Talc, or Mineral Granules; also Asphalt Shingles Surfaced with Mineral Granules (D 228 - 33 T), with certain editorial changes presented on the floor at the annual meeting, and for Coarse Particles in Mixtures of Asphalt and Mineral Matter (D 313 - 35 T) were accepted, the specifications and methods as revised being continued as tentative.

It was announced that the committee had completed the preparation of a new proposed Method of Testing Films Deposited from Bituminous Emulsions which, if approved by letter ballot of the committee, would be submitted to the Society for publication as tentative through Committee E-10 on Standards, subsequent to the annual meeting.

The following papers were then presented and discussed:

"The Constitution of Cracked and Uncracked Asphalts," by E. S. Hillman and B. Barnett (presented by Mr. Barnett).

"Development of Internal Structure in Asphalts with Time," by R. N. Traxler and C. E. Coombs (presented by Mr. Traxler).

"Indentation and Compression Shear Tests for Determining Service Stability of Asphalt Plank," by H. W. Greider and Henri Marc (presented from manuscript by Mr. Marc).

"The Design of Asphalt Mixtures for Underwater Construction," by Rossiter M. McCrone and F. C. Field (presented by Prévost Hubbard).

The meeting then adjourned till the following morning.

SEVENTH SESSION—WEDNESDAY, JUNE 30, 9.30 A. M.

Road Materials, Bituminous Materials

(Continued from Sixth Session; Held Simultaneously with Eighth and Ninth Sessions)

Mr. C. S. Reeve in the chair.

The report of Committee D-4 on Road and Paving Materials was presented by the First Vice-chairman, E. F. Kelly.

The proposed Tentative Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine was accepted for publication as tentative with an editorial change presented on the floor at the annual meeting to provide for the use of either cast iron or steel spheres as the abrasive charge in the test. The proposed Tentative Specifications for Standard Sizes of Coarse Aggregates for Highway Construction were accepted for publication as tentative to replace the existing Tentative Specifications D 63 - 23 T and D 64 - 20 T.

The revisions proposed for immediate adoption in the Standard Specifications for Materials for Cement Mortar Bed for Brick, Stone Block, Wood Block, Asphalt Block and Other Block Pavements (D 58 – 24) were unanimously approved for reference to letter ballot of the Society for adoption as standard, this recommendation requiring a nine-tenths vote.

The proposed revision of the Tentative Method of Test for Abrasion of Gravel (D 289 – 28 T) was accepted with the following additional revision in Section 3 presented on the floor at the annual meeting, the method as revised being continued as tentative:

Section 3.—Change from its form as preprinted: namely,

> 3. An abrasive charge weighing 2500 g. ± 10 g., comprising six cast-iron spheres approximately $1\frac{7}{8}$ in. in diameter, each weighing between 405 and 435 g., shall be used with each test sample.

to read as follows:

> 3. An abrasive charge weighing 2500 g. ± 10 g., comprising six cast-iron or steel spheres approximately $1\frac{7}{8}$ in. in diameter, each weighing between 405 and 440 g., shall be used with each test sample. The cast-iron spheres shall conform to the following requirements as to chemical composition:

Combined carbon, min., per cent	2.50
Graphitic carbon, max., per cent	0.25
Silicon, max., per cent	1.00
Manganese, max., per cent	0.50
Phosphorus, max., per cent	0.25
Sulfur, max., per cent	0.08

The Tentative Method of Test for Determination of Amount of Material Finer than No. 200 Sieve in Aggregates (C 117 – 35 T) was approved for reference to letter ballot of the Society for adoption as standard, on the recommendation presented jointly by Committee D-4 and Committee C-9 on Concrete and Concrete Aggregates, to replace the existing Standard Methods of Test for Quantity of Clay and Silt in Gravel for Highway Construction (D 72 – 21) and of Decantation Test for Sand and Other Fine Aggregates (D 136 – 28), the withdrawal of which standard methods was accordingly approved.

The withdrawal of the Standard Form of Specifications for Certain Commercial Grades of Broken Stone (D 35 – 18) was approved.

The following editorial changes in the Standard Methods of Chemical Analysis of Calcium Chloride (D 345 – 34) presented on the floor at the annual meeting, were approved:

Section 1.—Change the tenth sentence from its present form: namely,

> Dissolve the precipitate with 10 to 15 ml. of 0.25 N H_2SO_4, heat to about 80 C. and titrate to a pink color with 0.1 N $KMnO_4$.

to read as follows:

> Dissolve the precipitate with 10 to 15 ml. of a solution of 1 part H_2SO_4 and 4 parts water, heat to about 80 C. and titrate to a pink color with 0.1 N $KMnO_4$.

In the formula following this section change the denominator of the fraction to read as follows by the addition of the italicized word: "Weight of *original* sample in grams."

Section 3.—Divide the second paragraph of this section into two paragraphs beginning the new third paragraph with the present sixth sentence which reads: "Finally, filter through a dry retentive filter paper, discarding the first 10 ml. and refiltering if cloudy."

Omit from the new third paragraph the second sentence, which reads as follows: Add a few milliliters of $(NH_4)_2C_2O_4$, heat to boiling, and filter.

The recommendation, presented on the floor at the annual meeting, that the Tentative Specifications for Concrete for Pavements (D 366 – 33 T) be withdrawn, was approved. In explanation of this recommendation, it was pointed out that these specifications are, to a great extent, construction specifications for concrete pavements, and since there has been some question as to whether the Society should concern itself with specifications of this character, it was agreed that the specification would be discontinued with the understanding that this subject would be carried on by the American Concrete Institute.

It was announced that the committee had completed certain revisions in the Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (D 61 – 24) which, after approval by letter ballot of the committee, would be submitted to the Society through Committee E-10 on Standards during the summer for publication as tentative.

The following papers were then presented:

"Compression Testing of Asphalt Paving Mixtures—II," by Roland Vokac, presented by the author and discussed.

"Bituminous Practice on Western Highways,"[1] by J. E. Buchanan, presented from manuscript by B. E. Gray.

"The Homogeneity of West Texas Asphalts," by Sidney Born, presented from manuscript by the author and discussed.

"The Constituents of Asphaltic Materials *versus* Accelerated Weathering," by R. R. Thurston, presented by the author and discussed.

The meeting then adjourned till 1.30 p. m.

EIGHTH SESSION—WEDNESDAY, JUNE 30, 9.30 A. M.

Masonry Materials, Refractories

(Held Simultaneously with Seventh and Ninth Sessions)

Past-President Cloyd M. Chapman in the chair.

The report of Committee C-3 on Brick was presented by the chairman, J. W. McBurney. In presenting this final report of Committee C-3 Mr. McBurney announced that the proposal to merge Committee C-3 and Committee C-10 on Hollow Masonry Building Units as a single standing committee on manufactured masonry units, had been approved by letter ballot of the committees.

The proposed Tentative Specifications for Paving Brick (C 7 – 37 T) were accepted for publication as tentative to replace the Standard Specifications for Paving Brick (C 7 – 30); also the proposed Tentative Specifications for Sewer Brick (Made from Clay or Shale) (C 32 – 37 T) were accepted for publication as tentative to replace the Standard Specifications for Clay Sewer Brick (C 32 – 24). The withdrawal of the Standard Specifications C 7 – 30 and C 32 – 24 was accordingly approved.

The proposed revisions of the Tentative Specifications for Building Brick (Made from Clay or Shale) (C 62 – 36 T) were accepted, these specifications as revised being continued as tentative.

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[1] Published in ASTM BULLETIN, October, 1937.

The revisions proposed for immediate adoption in the Standard Specifications for Concrete Building Brick (C 55 – 34), and for Sand-Lime Building Brick (C 73 – 30) were unanimously approved for reference to letter ballot of the Society for adoption as standard, this recommendation requiring a nine-tenths vote. The Tentative Methods of Testing Brick (Modulus of Rupture, Compressive Strength, Absorption) (C 67 – 36 T) were approved for reference to letter ballot of the Society for adoption as standard to supersede the existing Standard Methods of Testing Brick (Compression, Flexure, Absorption) (C 67 – 31), which were accordingly withdrawn.

A paper entitled "The Wick Test for Efflorescence of Building Brick," by J. W. McBurney and D. E. Parsons, was presented by Mr. McBurney and discussed.

The report of Committee C-11 on Gypsum was presented by the chairman, J. W. Ginder.

An informal progress report of Sectional Committee A42 on Specifications for Plastering was presented by the Secretary, J. W. McBurney. It was announced that the committee had completed the preparation of specifications for gypsum plastering which, if approved by letter ballot of the committee, would be submitted to the Society for publication as tentative through Committee E-10 on Standards subsequent to the annual meeting.

The report of Committee C-8 on Refractories was presented by the chair. The proposed Tentative Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes, and the Definitions of Terms Relating to Alumina-Diaspore Refractories were accepted for publication as tentative. The Proposed Tentative Methods of Chemical Analysis of Refractory Materials (C 18 – 37 T) were accepted for publication as tentative to replace the Standard Methods of Ultimate Chemical Analysis of Refractory Materials (C 18 – 35), which were accordingly withdrawn.

The Tentative Method of Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 – 36 T) was approved for reference to letter ballot of the Society for adoption as standard.

The tentative revisions of the Standard Definitions for Fireclay Refractories (C 27 – 35) were unanimously approved for reference to letter ballot of the Society for adoption as standard, this recommendation requiring a nine-tenths vote. The recommendation for adoption as standard of the Tentative Definitions of Terms Relating to Refractories (C 71 – 36 T) was discussed. It was announced that consideration had been given to these definitions as required by the regulations of the Society by Committee E-8 on Nomenclature and Definitions. As a result of certain questions concerning the definitions, the Advisory Committee of Committee E-8 asked that action on the adoption of the definitions be deferred, and on motion, the definitions were continued as tentative.

A paper entitled "The Celite Type High-Temperature Thermal Conductivity Apparatus," by C. E. Weinland, was presented by G. A. Downsbrough.

The report of Committee C-10 on Hollow Masonry Building Units was presented by the chairman, D. E. Parsons. In presenting this report Mr. Parsons announced that the proposal to merge Committee C-10 with Committee C-3 on Brick as a single standing committee on manufactured masonry units, had been approved by letter ballot of the committees.

The proposed Tentative Specifications and Methods of Test for Concrete Units for Non-Load Bearing Masonry were accepted for publication as tentative.

A verbal progress report of Committee C-18 on Natural Building Stones and Slate was presented by the secretary, H. S. Brightly.

The meeting then adjourned till 1.30 p. m.

NINTH SESSION—WEDNESDAY, JUNE 30, 9.30 A. M.

Fatigue, Effect of Temperature

(Held Simultaneously with Seventh and Eighth Sessions)

Vice-President A. E. White in the chair.

The report of the Joint Research Committee on Effect of Temperature on the Properties of Metals was presented by the chairman, H. J. French. The proposed revisions of the Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 - 34 T), comprising changes in the tension test as given in Appendix I of the methods and the inclusion of an addendum on preparation of cast tension test specimens of heat-resistant metals were accepted, the method as revised being continued as tentative. In presenting the report Mr. French called upon J. G. Lowther to present the "Report on Long-Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel and 0.35 per cent Carbon Steel," as a report to the Joint Committee covering a sponsored research project carried out at Battelle Memorial Institute. Mr. French also called upon J. J. Kanter to present the paper by H. W. Gillett on "Discrepancies in the Load-Carrying Abilities of Carbon Steels at 850 F.," which after presentation was discussed.

The following papers were then presented by the respective authors and discussed:

"A Comparison of the Methods Used for Interpreting Creep Test Data," by Joseph Marin.

"New Equipment for Creep Tests at Elevated Temperatures," by P. G. McVetty.

"The Relaxation of Copper at Normal and at Elevated Temperatures," by John Boyd.

Mr. H. H. Morgan then assumed the chair.

The report of the Research Committee on Fatigue of Metals was presented by the chairman, H. F. Moore, and discussed.

A paper entitled "A Fatigue Machine for Testing Metals at Elevated Temperatures," by F. M. Howell and E. S. Howarth, was presented by Mr. Howarth and discussed.

A paper entitled "Fatigue Properties of Metals Used in Aircraft Construction at 3450 and 10,600 cycles," by T. T. Oberg and J. B. Johnson, was presented by R. L. Templin and discussed.

The meeting then adjourned till 1.30 p. m.

TENTH SESSION—WEDNESDAY, JUNE 30, 1.30 P. M.

Steel, Wrought Iron, Metallography

(Held Simultaneously with Eleventh Session)

Mr. N. L. Mochel in the chair.

The report of Committee A-2 on Wrought Iron was presented by the chairman, J. B. Young. The proposed revisions of the Tentative Specifications for Wrought-Iron Plates (A 42 - 36 T), for Wrought-Iron Rivets and Rivet Rounds (A 152 - 36 T) and for Single and Double Refined Wrought-Iron Bars (A 189 – 36 T) were accepted, the specifications as revised being continued as tentative. The proposed revisions of the Standard Specifications for Iron and Steel Chain (A 56 – 30), for Welded Wrought-Iron Pipe (A 72 – 33), for Wrought-Iron Rolled or Forged Blooms and Forgings (A 73 – 36), for Staybolt Wrought Iron, Solid (A 84 – 36), for Staybolt Wrought Iron, Hollow Rolled (A 86 – 36), for Uncoated Wrought-Iron Sheets (A 162 – 36) and for Zinc-Coated (Galvanized) Wrought-Iron Sheets (A 163 – 36) were accepted for publication as tentative.

It was announced that the committee had completed the preparation of new specifications for structural wrought-iron shapes and bars, and had prepared tentative revisions of the Standard Specifications for Iron and Steel Chain (A 56 – 30), for Staybolt Wrought Iron, Solid (A 84 – 36) and for Staybolt Wrought Iron, Hollow Rolled (A 86 – 36), which, if approved by letter ballot of the committee would be submitted to the Society for publication as tentative through Committee E-10 on Standards subsequent to the annual meeting.

The report of Committee A-1 on Steel was presented by the chairman, H. H. Morgan. The proposed Tentative Specifications for Seamless Intermediate Alloy Steel Still Tubes for Refinery Service, for Seamless Cold-Drawn Intermediate Alloy Steel Heat-Exchanger and Condenser Tubes, for Carbon-Silicon-Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels, for Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels, for Low-Carbon-Nickel Steel Plates for Boilers and Other Pressure Vessels, and for Molybdenum-Steel Plates for Boilers and Other Pressure Vessels, were accepted for publication as tentative.

The proposed Tentative Specifications for Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods), which were prepared jointly by the American Welding Society and the A.S.T.M., were accepted for publication as tentative with the following change presented on the floor at the annual meeting:

Section 14 (*a*).—Change from the form as preprinted to read as follows by the omission of the words in brackets:

(*a*) [A.S.T.M. specification number and] Grade.

The proposed revisions of the Standard Specifications for Structural Rivet Steel (A 141 – 36), for High Tensile Strength Carbon-Steel Plates for Pressure Vessels (Plates 2 in. and Under in Thickness) (A 149 – 36) and for High Tensile Strength Carbon-Steel Plates for Fusion-Welded Pressure Vessels (Plates over 2 in. to 4 in., inclusive, in Thickness) (A 150 – 36) were accepted for publication as tentative.

The Tentative Specifications for Fabricated Steel Bar or Rod Mats for Concrete Reinforcement (A 184 – 36 T), for Welded Steel Wire Fabric for Concrete Reinforcement (A 185 – 36 T), for Forged or Rolled Steel Pipe Flanges for General Service (A 181 – 35 T), for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 35 T), for Seamless Low-Carbon-Steel Still Tubes for Refinery Service (A 161 – 35 T) and for Cold-Drawn-Steel Heat-Exchanger and Condenser Tubes (A 179 – 35 T) were approved for reference to letter ballot of the Society for adoption as standard. The withdrawal of the List of Specifications for Steel Suitable for Fusion Welding (A 151 – 35) was approved.

It was announced that the committee had completed the preparation of a revision of the existing Tentative Specifications for Alloy-Steel Bolting Materials for High Pressure and Temperatures up to 1100 F. (A 193 – 36 T); also that it was planned to revise and recommend the reversion to tentative of the Standard Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F., including the submission as a new tentative specification for pipe of the carbon-molybdenum grade of steel, at present covered by these standard specifications. These recommendations, after approval by letter ballot of the committee, will be submitted to the Society for publication as tentative through Committee E-10 on Standards subsequent to the annual meeting.

The report of the Research Committee on Yield Point of Structural Steel was presented by the chairman, M. O. Withey. Since this was the final report on the work of the research committee, on motion it was discharged with a vote of thanks.

The following papers were presented by the respective authors and discussed:

"Steel Structures Identified and Flaws Located by Means of Balancing Wave Tests," by Carl Kinsley.

"Weld Metal as an Engineering Material and Some Methods of Testing," by L. J. Larson.

"A Fundamental Study of the Design of Impact Test Specimens," by H. C. Mann (presented from manuscript).

The report of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys was presented by the chairman, Jerome Strauss.

The report of Committee E-4 on Metallography was presented by the chairman, J. T. Norton.

The meeting then adjourned till 4.15 p. m.

ELEVENTH SESSION—WEDNESDAY, JUNE 30, 1.30 P. M.

Petroleum Products, Electrical Insulating Materials, Rubber, Soap, Textiles

(Held Simultaneously with Tenth Session)

Vice-President T. G. Delbridge in the chair.

The report of Committee D-2 on Petroleum Products and Lubricants was presented by the chairman, T. A. Boyd. In presenting the report Mr. Boyd called on Mr. Geniesse to present the paper entitled "Method for Converting Kinematic Viscosity to Saybolt Universal Viscosity," by C. E. Headington and J. C. Geniesse, which appeared as an appendix to the report.

The proposed Method of Test for Dropping Point of Lubricating Greases was accepted for publication as information. The proposed Method of Test for Unsulfonated Residue of Plant Spray Oils, with the following changes presented on the floor at the annual meeting subject to favorable letter ballot of Committee D-2,[1] was accepted for publication as information:

Section 2 (*a*).—Change from the form as preprinted to read as follows by the addition of the italicized words:

> 2. (*a*) Sulfuric Acid (37 N).—Prepare a 37 N sulfuric acid solution by blending reagent-grade fuming and concentrated sulfuric acids to 98.61 per cent ± 0.2 per cent H_2SO_4 at a temperature of 59 F. (15 C.), *to be determined by accurate titration.*

Section 3.—In Paragraph (*a*) of this section, change the word "standard" to read "ordinary" in describing the glass bottles used for holding the samples of oil. Change Paragraph (*b*) of this section to read as follows by the addition of the italicized words and the omission of the word in brackets:

> (*b*) Pipette.—One 5-ml. pipette, *precision grade,* [especially] calibrated *in the laboratory* to deliver 5 ml. ± 0.005 ml. of oil at 77 F.

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[1] This recommendation was submitted to letter ballot of Committee D-2 which consists of 84 members; 70 members returned their ballots, of whom 49 voted affirmatively and 2 negatively.

Change the first two sentences of Paragraph (*d*) of this section from the form as preprinted to read as follows by the addition of the italicized words and figures:

The test bottles shall be made of a good quality glass and *the preferred form* shall conform to the dimensions given in Fig. 1. The capacity to base of neck shall be 45 ml. ± 1.0 ml. *As an alternative, the standard* $6\frac{1}{2}$*-in. 50-per-cent cream bottle may be used provided the same accuracy is maintained.*

Change the next to last sentence of Paragraph (*d*) from the form as preprinted to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

The graduation marks shall be clear and fine, not more than [0.1 mm. in thickness] *0.3 mm. in width.*

In Paragraph (*f*) of this section, change the second sentence from the form as preprinted to read as follows by the addition of the italicized words and the omission of the words and figures in brackets:

The preferred form of centrifuge shall have a diameter of swing (at the base of whirling bottles) [of 15 to 17 in.] *sufficient to accommodate the test bottles without danger of interference when placed in opposite carriers*, and a speed of 1000 r.p.m.

In Paragraph (*g*) of this section change the expression "a mechanical shaker" to read "A preferred form of mechanical shaker."

Section 4.—In the description of the time of operation of the mechanical shaker in Paragraph (*d*) change the expression "for exactly 10 sec." to read "for 10 sec."

In Paragraph (*f*) of this section change the first sentence from the form as preprinted to read as follows by the addition of the italicized words:

The test bottle shall then be removed from the water bath, and sufficient sulfuric acid (sp. gr. 1.84) added to raise the liquid level in the neck of the bottle to the 50 per cent mark, *that is, the top graduation.*

The proposed Tentative Methods of Test for Kinematic Viscosity, with the following changes presented on the floor at the annual meeting subject to favorable letter ballot of Committee D-2,[1] were accepted for publication as tentative:

Section 2 (*b*).—Under the details of the thermometer requirements, change the maximum scale error from "0.05 F." to read "0.10 F."; also change the paragraph on Standardization from the form as preprinted to read as follows by the addition of the italicized words and figures:

STANDARDIZATION: The thermometers are to be standardized at the ice point, and at the viscosity point (100, 130, or 210 F.) for conditions of total immersion, *expressed to the nearest 0.02 F. and such corrections are to be added to the observed reading.*

Fig. 1.—In the diagram of the suspended level viscosimeters designate the upper and lower detail drawings as "square shoulder" and "spherical shoulder," respectively.

Section 3 (*a*).—Change the expression "current of air" to read "current of clean dry air."

Section 4.—Change the definition of *B* as used in Eq. 1 from the form as preprinted to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

B = an experimental constant determined by the design of the viscosimeter. For [Ubbelohde] viscosimeters with capillary diameters of 0.75 mm. or larger, the value of this constant is 2.0 *for Ubbelohde design and 1.0 for FitzSimons design;*

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[1] This recommendation was submitted to letter ballot of Committee D-2 which consists of 84 members; 70 members returned their ballots, of whom 66 voted affirmatively and 0 negatively.

in the case of viscosimeters having capillary diameters less than 0.75 mm., the value of the constant shall be determined in accordance with Section 5 (*b*). [For FitzSimons' modification the constant is 1.0.]

Section 5.—In Paragraph (*b*) of this section change the second sentence from the form as preprinted to read as follows by the addition of the italicized words:

In the case of the viscosimeter with square-shouldered outlet tube, the mean effective head varies with liquids of different surface tension.

Add a new paragraph at the end of this section to read as follows:

If a viscosimeter with spherical shoulders is used, the head does not vary with surface tension; therefore the multiplying constant is 1.000 instead of 1.0035.

The proposed Tentative Method of Test for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity was accepted for publication as tentative.

The proposed Tentative Method of Test for Distillation of Plant Spray Oils, with the following change presented on the floor at the annual meeting subject to favorable letter ballot of Committee D-2,[1] was accepted for publication as tentative:

Section 9 (*f*).—In the first sentence of this section change the expression "at a constant temperature of 140 F. (60 C.)" to read "at a temperature of approximately 140 F. (60 C.)."

The proposed Tentative Method of Test for Vapor Pressure of Petroleum Products (Reid Method), which represents a revision and consolidation of the existing Tentative Methods of Test for Vapor Pressure of Natural Gasoline (Reid Method) (D 323 – 32 T) and Test for Vapor Pressure of Motor and Aviation Gasoline (Reid Method) (D 417 – 35 T), was accepted for publication as tentative.

The proposed revision of the Tentative Method of Test for Knock Characteristics of Motor Fuels (D 357 – 36 T) was accepted, the method as revised being continued as tentative.

The proposed revision of the Tentative Standard Viscosity-Temperature Chart for Liquid Petroleum Products (D 341 – 32 T), which recommendation appeared in the report as preprinted, covering a viscosity range of 33 to 100,000,000 Saybolt Universal seconds and a temperature range of –30 to +450 F., was accepted. In addition the committee presented on the floor at the annual meeting a recommendation for the following three additional viscosity-temperature charts of the ranges and temperatures indicated, which were accepted for publication as tentative, subject to favorable letter ballot of Committee D-2:[2]

Abridged Saybolt Chart (8½ by 11 in. in size): Viscosity range of 33 to 100,000 seconds and a temperature range of –10 to +300 F.

Kinematic Chart: Viscosity range of 2 to 20,000,000 centistokes and a temperature range of –30 to +450 F.

Kinematic Chart: Viscosity range of 0.4 to 100 centistokes and a temperature range of –30 to +450 F.

The revisions presented for immediate adoption in the Standard Methods of Analysis of Grease (D 128 – 27) and Test for Melting Point of Paraffin Wax (D 87 – 22) were unanimously approved for reference to letter ballot of the Society for adoption as standard, this recommendation requiring a nine-tenths vote.

The recommendation presented on the floor at the annual meeting that the following revisions be adopted in the Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (D 287 – 36) was unanimously approved for reference to letter ballot of the Society, subject to favorable letter ballot of Committee D-2:[1]

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[1] This recommendation was submitted to letter ballot of Committee D-2 which consists of 84 members; 70 members returned their ballots, of whom 50 voted affirmatively and 0 negatively.

[2] This recommendation was submitted to letter ballot of Committee D-2 which consists of 84 members; 70 members returned their ballots, of whom 68 voted affirmatively and 1 negatively.

Section 3.—Change the second sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> It shall be of glass of the conventional constant mass and variable displacement type, with a [paper hand-written] scale *of suitable material* [printed in] *clearly marked with* black ink.

In the eighth and ninth sentences omit the word "paper" in the description of the hydrometer scale; also in the eighth sentence, substitute the word "marked" for "printed."

Section 4 (*b*).—Change this section to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> (*b*) Thermometer for Thermo-hydrometer.—The thermometer incorporated in the thermo-hydrometer type of instrument shall [be of the paper scale type (scale to be placed in either the bulb or stem of the hydrometer) having] *have* a range of from 20 to 130° F. or 60 to 220° F. depending upon the range of the hydrometer. The [paper] temperature scale shall be graduated in 2° F. divisions accurate to ±1° F. (Note) and, when located in the stem of the hydrometer, shall be [printed] *marked* in red ink to avoid any possibility of its being confused with the black gravity scale.

The recommendation presented on the floor at the annual meeting that the following revisions in the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (D 88 – 36) be accepted for publication as tentative was unanimously approved, subject to favorable letter ballot of Committee D-2:[2]

Section 4 (*c*).—Change the note following this section to read as follows by the addition of the italicized sentence:

> NOTE.—The plunger commonly supplied with the viscosimeter shall never be used on instruments maintained as standards. *In case the flash point is less than 50 F. above the temperature of test, the wetting operation should be omitted in order to avoid an increase in viscosity due to evaporation of a portion of the light fraction.*

Section 4 (*d*).—After the first sentence add a new sentence to read as follows:

> In case the flash point is less than 50 F. (10 C.) above the temperature of test, the oil shall be heated to room temperature only.

A recommendation presented on the floor at the annual meeting for the publication in the 1937 edition of the compilation of A.S.T.M. Standards for Petroleum Products and Lubricants of the Diesel Fuel Oil Classification, revised by the addition of a provision that oils Nos. 1D, 3D, 4D and 5D shall be free from alkali and inorganic acid, was approved subject to favorable letter ballot of Committee D-2.[3]

It was also announced that the committee had approved editorial changes in the Standard Methods of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 – 35), Test for Distillation of Natural Gasoline (D 216 – 32), Test for Burning Quality of Kerosine Oils (D 187 – 36), Test for Burning

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[1] This recommendation was submitted to letter ballot of Committee D-2 which consists of 84 members; 70 members returned their ballots, all of whom voted affirmatively.

[2] This recommendation was submitted to letter ballot of Committee D-2, which consists of 84 members; 70 members returned their ballots, of whom 68 voted affirmatively and 2 negatively.

[3] This recommendation was submitted to letter ballot of Committee D-2 which consists of 84 members; 70 members returned their ballots, of whom 61 voted affirmatively and 1 negatively.

Quality of Mineral Seal Oil (D 239 – 30), Test for Dilution of Crankcase Oils (D 322 - – 35) and Tentative Method of Test for Sulfur in Petroleum Oils by Lamp Method (D 90 – 34 T).

The report of Sectional Committee Z11 on Petroleum Products and Lubricants was presented from manuscript by the chairman, T. A. Boyd.

A paper entitled "A Laboratory Channel Test for Gear Oils," by J. P. Stewart, was presented by the author and discussed.

An informal report of Committee D-15 on Thermometers and Laboratory Glassware was presented by the chairman, W. H. Fulweiler.

Past-President H. S. Vassar then assumed the chair.

The report of Committee D-11 on Rubber Products was presented by the chairman, O. M. Hayden. The proposed Method for Air Pressure Heat Test was accepted for publication as tentative with certain editorial changes presented on the floor at the annual meeting.

The proposed revisions of the Tentative Methods of Test for Abrasion Resistance of Rubber Compounds (D 394 – 34 T), Test for Compression Set of Vulcanized Rubber (D 395 – 36 T), Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 – 36 T), and for Insulated Wire and Cable: Performance Rubber Compound (D 353 – 36 T) were accepted, the methods and specifications as revised being continued as tentative. The withdrawal of the Tentative Specifications for Insulated Wire and Cable: Class A, 30 per cent Hevea Rubber Compound (D 393 – 34 T) was approved.

It was announced that the committee had completed the preparation of proposed Methods of Test for Hose which represented a revision and consolidation of the existing Standard Methods of Test for Rubber Hose (D 380 – 36) and (D 379 – 36); also that a tentative revision had been prepared of the Standard Specifications for Cotton Rubber-Lined Fire Hose for Public and Private Fire Department Use (D 296 – 35). The committee had also prepared new specifications for rubber gloves, a new method of test for change of properties of rubber in liquids, and changes in the wire specifications. These recommendations, if approved by letter ballot of the committee, would be submitted to the Society for publication as tentative through Committee E-10 on Standards subsequent to the annual meeting.

The report of Committee D-12 on Soaps and Detergents was presented by the chairman, H. B. Trevithick. The proposed Methods of Chemical Analysis of Sulfonated (Sulfated) Oils with editorial changes, and of Chemical Analysis of Special Detergents were accepted for publication as information.

The proposed Tentative Specifications for Milled Toilet Soap, for Caustic Soda, for Modified Soda (Sesquicarbonate Type), for Soda Ash, Methods of Sampling and Chemical Analysis of Soaps and Soap Products with editorial changes, and Definitions of Terms Relating to Soaps and Detergents were accepted for publication as tentative.

The report of Committee D-13 on Textile Materials was presented by the chairman, H. J. Ball. The proposed Tentative Methods of Test for Wool Felt, Testing and Tolerances for Certain Wool and Part Wool Fabrics and Test for Volumetric Determination of Small Amounts of Copper in Textiles were accepted for publication as tentative. The proposed new Standard Specifications and Methods of Test for Tire Fabrics Other than Cord Fabrics recommended for immediate adoption as a revision of and to replace the existing Standards D 122 – 33, D 316 – 34 and D 336 – 33 were unanimously approved for reference to letter ballot of the Society for adoption as standard, this recommendation requiring a nine-tenths vote. The withdrawal of the Standard Methods of Testing and Tolerances for Tire Fabrics Other than Cord

Fabrics (D 122 – 33), Specifications and Methods of Test for Chafer Tire Fabrics (D 316 – 34) and for Enameling Duck for the Tire Industry (D 336 – 33) was approved.

The proposed revisions of the Tentative Definitions and Terms Relating to Textile Materials (D 123 – 36 T), Methods of Testing and Tolerances for Rayon (D 258 - 35 T), Methods for the Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles (D 276 – 35 T), General Methods of Test for Properties of Cotton Fibers (D 414 – 35 T) and Methods of Testing Pile Floor Covering (D 418 – 36 T) were accepted, the definitions and methods of test as revised being continued as tentative.

The proposed revision of the Standard General Methods of Testing Woven Textile Fabrics (D 39 – 36) was accepted for publication as tentative. The revisions proposed for immediate adoption in the Standard Specifications for Textile Testing Machines (D 76 – 36), General Methods of Testing and Tolerances for Cotton Yarns and Threads (D 180 – 36), Specifications and Methods of Test for Asbestos Yarns (D 299 – 36), for Asbestos Roving for Electrical Purposes (D 375 – 36) and for Asbestos Tape for Electrical Purposes (D 315 – 36) were unanimously approved for reference to letter ballot of the Society for adoption as standard, this recommendation requiring a nine-tenths vote.

The Tentative Methods of Test for Fineness of Wool (D 419 – 35 T), Test for Fastness of Dyed or Printed Cotton Fabrics to Laundering or Domestic Washing (D 435 – 36 T), Test for Fastness of Dyed or Printed Silk or Rayon Fabrics to Laundering or Domestic Washing (D 436 – 36 T) and Test for Small Amounts of Copper and Manganese in Textiles (D 377 – 36 T), with revisions in the latter three methods, were approved for reference to letter ballot of the Society for adoption as standard.

The report of Committee D-9 on Electrical Insulating Materials was presented by the chairman, T. Smith Taylor. The proposed revisions of the Standard Methods of Testing Electrical Porcelain (D 116 – 34) and Test for Resistivity of Insulating Materials (D 257 – 33), the latter in the form of extensively revised methods, were accepted for publication as tentative.

The proposed revisions of the Tentative Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 – 36 T), Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 36 T), Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 – 36 T), Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 – 35 T), Testing Shellac Used for Electrical Insulation (D 411 – 35 T), Specifications for Flexible Varnished Tubing Used in Electrical Insulation (D 372 – 35 T) and for Black Bias-Cut Varnished Cloth Tape Used for Electrical Insulation (D 373 – 36 T) were accepted, the methods and specifications as revised being continued as tentative. The tentative revision of the Standard Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 33) was unanimously approved for reference to letter ballot of the Society for adoption as standard, this recommendation requiring a nine-tenths vote.

A paper entitled "Determination of the Edge Correction in the Measurement of Dielectric Constant," by Arnold H. Scott, was presented by H. L. Curtis.

The meeting then adjourned till 4.15 p. m.

TWELFTH SESSION—WEDNESDAY, JUNE 30, 4.15 P. M.

Edgar Marburg Lecture

President Arno C. Fieldner in the chair.

President Fieldner in opening the session devoted to the Edgar Marburg Lecture summarized briefly the purpose and double significance of the lecture instituted in

1926, namely, to commemorate annually the name of the first Secretary-Treasurer of the Society, Dr. Edgar Marburg, and to bring to the Society valuable expositions on notable developments in engineering and the sciences. In paying a brief tribute to Edgar Marburg for his part in furthering the knowledge of engineering materials, President Fieldner referred particularly to his initiative and foresight at the time of the organization of the Society, which has made it such a valuable source for accurate technical information on the characteristics of engineering materials, made possible by continued research and investigative studies.

The President then introduced the twelfth Edgar Marburg Lecturer, Dr. T. Smith Taylor, Manager, Engineering Laboratory and Experimental Laboratory, Diehl Manufacturing Co., who had selected as the subject of the lecture "Plastics: Some Applications and Methods of Testing." Doctor Taylor presented a comprehensive survey of those materials which in industry are termed "plastics," including shellac, rubber, synthetic resins, casein products, and cellulose derivatives. In describing these newer materials which are finding increasing engineering applications, Doctor Taylor pointed out that plastics and plastic compounds occupy a very important place in everyday life and promise, through continued research, to be of ever-increasing value to civilization. He discussed the nature of such materials according to their origin, which divides them into two groups: natural and synthetic; also the types of plastics from the standpoint of the effects of heat upon them, namely, the thermoplastics or heat-non-convertible materials, and the thermo-setting or thermo-hardening substances. He considered in detail the nature, properties and applications of a number of plastic materials, such as rubber, shellac, cold-molded plastics, various types of cellulose plastics, including nitrate, and acetate, and ethyl cellulose plastics; also phenol-formaldehyde plastics, cast plastics, urea-formaldehyde plastics, furfural-phenol resins, vinyl resins, acrylic resin, cumarone resin, polystyrene, and casein plastics. He discussed briefly methods of testing these materials, with particular consideration to the development of the tension test for molded compounds. In discussing the subject of testing, a field in which much remains to be accomplished in the standardization of satisfactory procedures for obtaining authoritative information on the properties and availability of plastic materials, he advocated an intelligent interpretation as to the significance of test results on the part of both the consumer and the purchaser in the interest of obtaining improved and cheaper products.

President Fieldner in expressing appreciation to Doctor Taylor for his instructive and interesting lecture on a subject of timely interest to the Society, presented to him, on behalf of the Society, the Edgar Marburg Lecture Certificate.

President Fieldner then recognized W. H. Fulweiler, chairman of the Committee on Award of the Charles B. Dudley Medal. Mr. Fulweiler presented as the recipient of the 1937 Award of the Dudley Medal Messrs. W. H. Swanger and G. F. Wohlgemuth for a paper under their joint authorship entitled "Failure of Heat-Treated Steel Wire in Cables of the Mt. Hope, R. I., Suspension Bridge," presented at the 1936 annual meeting of the Society and considered by the committee as a paper of outstanding merit constituting an original contribution on research in engineering materials.

On behalf of and as the representative of the Society, President Fieldner then made the eleventh award of the Charles B. Dudley medal to Messrs. Swanger and Wohlgemuth.

In accepting the medal on behalf of Mr. Wohlgemuth and himself, Mr. Swanger expressed appreciation of the award as follows:

> "Mr. Wohlgemuth and I wish to express our sincere appreciation of the honor conferred by the award of the Charles B. Dudley Medal. Although personally the recipients of these medals, we feel that the award is a valued recognition of

the work accomplished by the organization of which we are members. In accepting this award we make grateful acknowledgment of the active cooperation and helpful advice received from many members of the staff of the National Bureau of Standards and others concerning the problem, mention of whom has been made in our paper. We are highly gratified to be admitted into that distinguished group of those who have preceded us as recipients of the Charles B. Dudley Medal. We thank you."

THIRTEENTH SESSION—THURSDAY, JULY 1, 9.30 A. M.

Timber, Naval Stores, Fire Tests

(Held Simultaneously with Fourteenth and Fifteenth Sessions)

Past-President Hermann von Schrenk in the chair.

The report of Committee D-17 on Naval Stores was presented by the chairman, F. P. Veitch. The proposed Tentative Method of Test for Acid Number of Rosin, and Test for Saponification Number of Rosin were accepted for publication as tentative.

A paper entitled "The Composition of American Steam-Distilled Wood Turpentine and a Method for Its Identification," by T. C. Chadwick and S. Palkin, was presented from manuscript by Mr. Chadwick.

The report of Committee D-7 on Timber was presented by the chairman, Hermann von Schrenk. The proposed Tentative Method of Test for Tar Acids in Creosote and Creosote - Coal Tar Solutions was accepted for publication as tentative. The Tentative Specifications for Timber Piles (D 25 - 35 T) and for Structural Wood Joist and Plank, Beams and Stringers, Posts and Timbers (D 245 - 36 T) were approved for reference to letter ballot of the Society for adoption as standard.

A paper entitled "The Mechanical Properties of Balsa Wood," by Jasper O. Draffin and C. W. Muhlenbruch, was presented by Mr. Muhlenbruch and discussed.

The report of Committee C-5 on Fire Tests of Materials and Construction was presented by the chairman, R. P. Miller. The recommendation for publishing as tentative the proposed Specifications for Fire-Retardant Properties of Wood for Scaffolding and Shoring was discussed. A motion to refer these proposed specifications back to the committee for further consideration failed to carry. On motion the proposed specifications were then accepted for publication as tentative.

The meeting then adjourned till 2 p. m.

FOURTEENTH SESSION—THURSDAY, JULY 1, 9.30 A. M.

Cement and Concrete

(Held Simultaneously with Thirteenth and Fifteenth Sessions)

Mr. F. E. Richart in the chair.

The report of Committee C-1 on Cement was presented by the chairman, P. H. Bates. A motion for the acceptance as tentative of the proposed Specifications for Blended Cements failed to receive the necessary two-thirds vote of those voting at the session, the results of the vote being 15 affirmative, and 13 negative.

The proposed revisions of the Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 - 34 T) and the new procedures for determining manganese and phosphorus in cement for inclusion in the revised Tentative Method of Chemical Analysis of Portland Cement (C 114 - 35 T) were accepted, the methods as revised being continued as tentative.

The revisions proposed for immediate adoption in the Standard Specifications for Portland Cement (C 9 - 30) and for Natural Cement (C 10 - 09), the latter in the form of completely revised specifications, were unanimously approved for reference to letter ballot of the Society for adoption as standard, this recommendation requiring a nine-tenths vote. The adoption as standard of Sections 1 to 10, with editorial modifications, of the Tentative Method of Chemical Analysis of Portland Cement (C 114 - 35 T) was approved for reference to letter ballot of the Society.

A paper entitled "Tricalcium Aluminate and the Microstructure of Portland Cement Clinker," by Levi S. Brown, was presented by the author.

The report of Committee C-9 on Concrete and Concrete Aggregates was presented by the vice-chairman, Stanton Walker.

The proposed Tentative Specifications for Lightweight Aggregate for Concrete were accepted for publication as tentative, with the following additional changes presented on the floor at the annual meeting, subject to favorable letter ballot of Committee C-9:[1]

Section 2 (*a*).—Change from the form as preprinted to read as follows by the addition of the italicized words:

2. (*a*) Lightweight aggregates shall consist of pumice, lava, tufa, slag, burned clay, *burned shale*, cinders derived from the high-temperature combustion of coal or coke showing a loss on ignition of not more than 25 per cent (40 per cent) and volatile matter of not more than 5 per cent, or, subject to approval by the engineer, other material having strong, durable particles and conforming to the requirements of these specifications.

Section 4.—Change from its form as preprinted: namely,

4. Lightweight aggregate meeting the grading requirements of these specifications when tested in a dry condition shall conform to the following minimum weight requirements:

	UNIT WEIGHT, LB. PER CU. FT.
Fine aggregate	70
Coarse aggregate	55 (75)[a]

[a] See Note under Section 1.

to read as follows:

4. Lightweight aggregate meeting the grading requirements of these specifications when tested in a dry condition shall conform to the following weight requirements:

	UNIT WEIGHT, LB. PER CU. FT.
Fine aggregate, not more than	70
Coarse aggregate, not more than	55[a]

[a] Heavier aggregate may be permitted when lightweight aggregates meeting the above requirements are not economically available and the higher weight of concrete resulting from their use is permissible.

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[1] This recommendation was submitted to letter ballot of Committee C-9, which consists of 60 members; 41 members returned their ballots, of whom 38 voted affirmatively and 1 negatively.

Section 5 (a).—Change the first sentence from the form as preprinted to read as follows by the addition of the italicized words and figures:

5. (*a*) Lightweight aggregate shall be of such quality that when made into a mortar and subjected to the mortar strength test (Section 9 (*d*)) it shall develop a compressive strength *at 7 or 28 days* of not less than 70 per cent (or, for concrete not exposed to wear or weather, 60 per cent), of that developed by a mortar prepared in the same manner, with the same cement, and the graded Ottawa sand described in Paragraph (*b*).

The proposed Tentative Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine was accepted for publication as tentative with a change in Section 3 of the methods as preprinted to provide for the use of cast iron or steel spheres as the abrasive charge in the test, subject to favorable letter ballot of Committee C-9.[1] The proposed revision of the Tentative Specifications for Concrete Aggregates (C 33 - 36 T) was accepted, the specifications as revised being continued as tentative.

The proposed Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate which represents a revision and consolidation of the existing Tentative Methods of Test for Soundness of Fine Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 - 35 T) and for Soundness of Coarse Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 89 - 35 T), was accepted for publication as tentative with the following additional changes presented on the floor at the annual meeting, subject to favorable letter ballot of Committee C-9.[2]

Section 2 (c).—Change the Note following this section from its form as preprinted to read as follows:

NOTE.—No standard form of container is recommended since different practices will be most convenient in various laboratories. The committee has had presented to it data indicating that the type of container may affect the results in some degree, but these data are not sufficiently comprehensive to permit of specifying a particular type. Two general methods of immersion have been used as follows:

(*1*) Each sample placed in a water-tight container and covered with the sodium sulfate or magnesium sulfate solution.

(*2*) Each sample placed in a container with a wire mesh or perforated bottom (depending upon size of particles being tested) and immersed in a vat containing the sodium sulfate or magnesium sulfate solution. Care should be taken to prevent loss by flotation if uncovered containers are submerged. If nested sieves are used, a pan should be inserted between sieves to prevent loss of material from one sieve to another.

Section 3.—Change the Note following Paragraph (*a*) of this section from its form as preprinted to read as follows:

NOTE.—For the solution, 215 g. of anhydrous salt or 485 g. of the decahydrate per liter of water are sufficient for saturation at 22 C. However, since these salts are not completely stable and since it is desirable that an excess of crystals be present, the use of not less than 350 g. of the anhydrous salt or 500 g. of the hydrous salt per liter of water is recommended.

Change the Note following Paragraph (*b*) of Section 3 from its form as preprinted to read as follows:

NOTE.—For the solution, 350 g. of anhydrous salt or 725 g. of the heptahydrate per liter of water are sufficient for saturation at 23 C. However, since these salts are not completely stable, with the hydrous salt being the more stable of the two, and since it is desirable that an excess of crystals be present, it is recommended that the hydrous salt be used and in an amount of not less than 800 g. per liter of water.

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[1] This recommendation was submitted to letter ballot of Committee C-9, which consists of 60 members, 41 members returned their ballots, of whom 39 voted affirmatively and 0 negatively.

[2] This recommendation was submitted to letter ballot of Committee C-9, which consists of 60 members; 41 members returned their ballots, of whom 39 voted affirmatively and 1 negatively.

Section 6.—Change the last sentence from its form as preprinted to read as follows by the omission of the words in brackets:

The samples immersed in the [sodium sulfate] solution shall be maintained at a temperature of 21 C. ± 1 C. (70 F. ± 2 F.) for the 18-hr. period.

The proposed revisions of the Standard Methods of Making and Storing Compression Test Specimens of Concrete in the Field (C 31 – 33), Specifications for Curing Portland-Cement Concrete Slabs with Calcium-Chloride Admixture (C 82 – 34) and for Curing Portland-Cement Concrete Slabs by Surface Application of Calcium Chloride (C 83 – 36) were accepted for publication as tentative.

The proposed revisions of the Standard Specifications for Ready Mixed Concrete (C 94 – 35), with an editorial change presented on the floor at the annual meeting, were accepted for publication as tentative.

The Tentative Method of Test for Determination of Amount of Material Finer than No. 200 Sieve in Aggregates (C 117 – 35 T) was approved for reference to letter ballot of the Society for adoption as standard, on the recommendation presented jointly by Committee C-9 and Committee D-4 on Road and Paving Materials, to replace the existing Standard Methods of Test for Quantity of Clay and Silt in Gravel for Highway Construction (D 72 – 21) and of Decantation Test for Sand and Other Fine Aggregates (D 136 – 28), which standard methods were accordingly withdrawn.

The Tentative Method of Test for Determination of Voids in Coarse Aggregate for Concrete (Dry Rodded) (C 30 – 35 T) was approved for reference to letter ballot of the Society for adoption as standard.

The withdrawal of the Standard Methods of Test for Absorption by Aggregates for Concrete (Laboratory Determinations) (C 95 – 36) and Field Test for Absorption of Mixing Water by Aggregates for Concrete (C 96 – 36) was approved.

In presenting the report Mr. Walker called on H. W. Leavitt to present from manuscript a report on "A Study of Plastic Mortar Cubes," submitted by Subcommittee XI on Evaluation of Data, which was accepted for publication, subject to approval of Committee E-6 on Papers and Publications.

Mr. H. F. Gonnerman then assumed the chair.

The report of Committee C-13 on Concrete Pipe was presented by the chairman, E. F. Kelly. The Tentative Specifications for Reinforced-Concrete Culvert Pipe (C 76 – 35 T), as revised, were approved for reference to letter ballot of the Society for adoption as standard.

A paper entitled "Plastic Flow and Volume Changes of Concrete," by Raymond E. Davis, Harmer E. Davis and Elwood H. Brown, was presented from manuscript by Raymond E. Davis and discussed.

A paper entitled "Some Tests to Show the Effect of Freezing on the Permeability, Strength, and Elasticity of Concretes and Mortars," by Herbert H. Scofield, was presented by title.

An informal report of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete in the absence of the chairman, A. E. Lindau, was presented by the Secretary-Treasurer, C. L. Warwick.

The meeting then adjourned till 2 p. m.

FIFTEENTH SESSION—THURSDAY, JULY 1, 9.30 A. M.

Corrosion

(Held Simultaneously with Thirteenth and Fourteenth Sessions)

Messrs. F. N. Speller and R. J. McKay, co-chairmen.

The report of Committee A-5 on Corrosion of Iron and Steel was presented by the chairman, F. F. Farnsworth. In presenting the report, Mr. Farnsworth called on R. F. Passano to present the report of Subcommittee VIII on Field Tests of Metallic Coatings, including a detailed report of the field tests on wire and wire products, and on L. W. Hopkins to describe the activities of Subcommittee VII on Methods of Testing.

The proposed revisions of the Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (A 116 - 30) and for Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (A 121 - 30), in the form of completely revised specifications, were accepted for publication as tentative.

The report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys was presented by the chairman, T. S. Fuller.

A paper entitled "Corrosion Testing Methods for Copper Alloys," by D. K. Crampton and N. W. Mitchell, was presented by Mr. Mitchell and discussed.

Mr. Speller before introducing the Discussion on Corrosion Testing Procedures, which was a continuation of that held in conjunction with the symposium on this subject at the Chicago Regional Meeting of the Society on March 2, called attention to the fact that this subject was of world-wide interest as evidenced by the considerations given to corrosion and corrosion testing at the conference in London at the meeting of the International Association for Testing Materials.

Mr. Speller then introduced L. W. Hopkins, who presented a Digest of the papers and discussions presented at the Chicago meeting in order to open the discussion on the subject. The meeting was then thrown open for general discussion, and the authors of the original papers were called upon for concluding remarks in response to the discussion submitted.

The meeting then adjourned till 2 p. m.

SIXTEENTH SESSION—THURSDAY, JULY 1, 2 P. M.

Water

(Held Simultaneously with Seventeenth Session)

Mr. A. H. White in the chair.

The report of Committee D-19 on Water for Industrial Uses was presented by the chairman, Max Hecht.

Mr. S. F. Acree then discussed "The Need for pH Standards," which subject was then opened for general discussion.

A paper entitled "Technique in the Determination of Dissolved Oxygen," by T. H. Daugherty, was presented by the author and discussed.

An informal progress report of the Joint Research Committee on Boiler Feedwater Studies[1] was presented from manuscript by the chairman, C. H. Fellows.

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[1] Published in ASTM BULLETIN, August, 1937.

A paper entitled "A Thermodynamic and Colloidal Interpretation of Published Studies on the Corrosion Cracking of Stressed Mild Steel in Water Solutions," by J. A. Tajc, was presented by the author and discussed.

A paper entitled "Determination of Hardness in Water by Direct Titration," by R. T. Sheen and C. A. Noll, was presented by Mr. Sheen and discussed.

A paper entitled "Some Applications of the Polarizing Microscope to Water-Conditioning Problems," by Everett P. Partridge, was presented by the author and discussed.

The meeting then adjourned till the following morning.

SEVENTEENTH SESSION—THURSDAY, JULY 1, 2 P. M.

Methods of Testing

(Held Simultaneously with Sixteenth Session)

Past-President H. F. Moore in the chair.

The report of Committee E-1 on Methods of Testing was presented by J. R. Townsend. A report on testing of thin sheet metals, presented on the floor at the annual meeting for publication, was accepted subject to favorable letter ballot of Committee E-1 and to approval by Committee E-6 on Papers and Publications.

It was announced that the committee had approved an editorial change in the title of the Tentative Method of Test for Softening Point by Ring-and-Ball Apparatus (E 28 – 36 T) changing this to read "Tentative Method of Test for Softening Point by Tapered Ring Apparatus." It was also announced that the committee had completed revised definitions for the terms consistency, plasticity and related properties, as a revision of the Tentative Definitions of Terms Consistency and Plasticity (E 24 – 34 T) which, if approved by letter ballot of the committee, would be submitted to the Society for publication as tentative through Committee E-10 on Standards subsequent to the annual meeting.

The following papers were then presented and discussed:

"A Classification of Methods of Mechanical Analysis of Particulate Materials," by Paul S. Roller presented by L. T. Work.

"The Cement Reference Laboratory,"[1] by J. R. Dwyer.

"Analysis of the Brinell Hardness Test," by R. H. Heyer.

"Measuring Elastic Drift," by Robert W. Carson.

"Improvement in the Adaptability of the Tuckerman Strain Gage," by L. A. Meisse.

"Speed Control for Screw-Power Testing Machines Driven by Direct-Current Motors,"[1] by A. H. Stang and L. R. Sweetman, presented by Mr. Stang.

"Dynamic Tests by Means of Induced Vibrations," by Rudolf K. Bernhard. The subject covered by the paper was illustrated by motion pictures.

The meeting then adjourned till the following morning.

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[1] Published in ASTM BULLETIN, August, 1937.

EIGHTEENTH SESSION—FRIDAY, JULY 2, 9.30 A. M.

Cast Iron

(Held Simultaneously with Nineteenth Session)

Mr. Hyman Bornstein in the chair.

The report of Committee A-3 on Cast Iron was presented by the secretary, H. C. Aufderhaar.

The report of Committee A-7 on Malleable Iron Castings was presented by the chairman, E. K. Smith.

The following papers were then presented and discussed:

"The Tensile Strength of Cast Iron," by J. O. Draffin and W. L. Collins, presented from manuscript by Mr Collins.

"A Study of the Effect of Span on the Transverse Test Results for Cast Iron," by J. T. MacKenzie and C. K Donoho, presented by Mr. Donoho.

"Relation of Properties of Cast Iron to Thickness of Castings," by H. L. Campbell, presented by J. T. MacKenzie.

"A Proposed Standard Classification of Graphite in Gray Cast Iron," by W. E. Mahin and J. W. Hamilton, presented by Mr. Mahin.

The meeting then adjourned.

NINETEENTH SESSION—FRIDAY, JULY 2, 9.30 A. M.

Non-Ferrous Metals

(Held Simultaneously with Eighteenth Session)

Mr. W. R. Webster in the chair.

The report of Committee B-1 on Copper and Copper Alloy Wires for Electrical Conductors was presented by the chair. The proposed revisions of the Tentative Specifications for Hard-Drawn Copper Alloy Wires for Electrical Conductors (B 105 - 36 T) were accepted, the specifications as revised being continued as tentative. The revisions proposed for immediate adoption in the Standard Specifications for Bronze Trolley Wire (B 9 - 36) and for Copper Trolley Wire (B 47 - 36) were unanimously approved for reference to letter ballot of the Society for adoption as standard, this recommendation requiring a nine-tenths vote.

The report of Committee B-2 on Non-Ferrous Metals and Alloys was presented by the secretary, E. E. Thum. The proposed revision of the Standard Specifications for Rolled Zinc (B 69 - 29) in the form of completely revised specifications were accepted for publication as tentative. The tentative revision of the Standard Specifications for Slab Zinc (Spelter) (B 6 - 33) was unanimously approved for reference to letter ballot of the Society for adoption as standard, this recommendation requiring a nine-tenths vote. A brief summary of the subcommittee activities of Committee B-2 was presented for inclusion in the report.

A paper entitled "Fatigue Properties of Non-Ferrous Sheet Metals," by C. H. Greenall and G. R. Gohn, was presented by Mr. Greenall and discussed.

The report of Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys was presented by the chairman, Dean Harvey, and accepted for publication subject to favorable letter ballot of the committee.[1] After presentation, the report was discussed. The proposed revision of the Tentative Method of Test for Flexivity of Thermoflex (Thermostatic Metals) (B 106 – 36 T) was accepted, the method as revised being continued as tentative, subject to favorable letter ballot of Committee B-4.[2]

A paper entitled "The Stiffness or Flexure Test," by H. L. MacBride, was presented by the author and discussed.

The report of Committee B-5 was presented by the secretary, D. K. Crampton. The proposed Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock were accepted for publication as tentative to replace the existing Standard Specifications B 44 – 33, B 56 – 33 and Tentative Specifications B 104 – 36 T. The withdrawal of the Standard Specifications for Seamless Admiralty Condenser Tubes and Ferrule Stock (B 44 – 33), for Seamless 70–30 Brass Condenser Tubes and Ferrule Stock (B 55 – 33), for Seamless Muntz Metal Condenser Tubes and Ferrule Stock (B 56 – 33) and Tentative Specifications for Seamless Copper-Nickel Alloy Condenser Tubes and Ferrule Stock (B 104 – 36 T) was approved. The proposed revision of the Tentative Specifications for Copper-Silicon Alloy Wire for General Purposes (B 99 – 36 T) was accepted, the specifications as revised being continued as tentative.

The report of Committee B-6 on Die-Cast Metals and Alloys was presented by the chairman, J. R. Townsend. The proposed revisions of the Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 – 33 T) and for Magnesium-Base Alloy Die Castings (B 94 – 34 T) were accepted, the specifications as revised being continued as tentative. A paper entitled "Brass Die Castings," by J. C. Fox, to appear as an appendix to the report of Committee B-6, was presented from manuscript by the author. A paper entitled "A Study of Die Design Changes for the Improvement of the Soundness and Uniformity of Test Bars," by G. L. Werley, which is also to appear as an appendix to the report, was presented from manuscript by the author.

The report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, was presented by the chairman, Sam Tour. In presenting the report Mr. Tour called attention to a report on "Modulus of Elasticity of Aluminum Alloys," by R. L. Templin and Sam Tour, to be appended to the report, and also a Report on Methods of Testing Oxide Coatings on Aluminum, by Junius D. Edwards, appended to the report.

The proposed Tentative Specifications for Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings were accepted for publication as tentative with the proposed deletion of Table II on precision requirements for methods of chemical analysis presented on the floor at the annual meeting, subject to favorable letter ballot of Committee B-7.[3] The proposed revision of the Tentative Specifications for Aluminum-Base Sand-Casting Alloys in Ingot Form (B 58 – 33 T) was accepted, the specifications as revised being continued as tentative, with the proposed deletion of Table II on precision requirements for methods of chemical analysis, presented on the floor at the annual meeting, subject to favorable letter ballot of Committee B-7.[1]

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[1] The report was submitted to letter ballot of Committee B-4 which consists of 44 members; 31 members returned their ballots, all of whom voted affirmatively.

[2] The proposed revision was submitted to letter ballot of Committee B-4 which consists of 44 members; 31 members returned their ballots, of whom 26 voted affirmatively and 0 negatively.

[3] This recommendation was submitted to letter ballot of Committee B-7, which consists of 41 members; 33 members returned their ballots, of whom 28 voted affirmatively and 0 negatively.

The recommendation presented on the floor at the annual meeting that the table of precision requirements for methods of chemical analysis expressed in percentage of the specified value appearing in the Tentative Specifications for Aluminum-Base Alloy Sand Castings (B 26 – 36 T) and for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 36 T) be deleted was unanimously approved, subject to favorable letter ballot of Committee B-7.[2]

The report of Committee E-2 on Spectrographic Analysis was presented from manuscript by the vice-chairman, T. A. Wright, and accepted for publication subject to favorable letter ballot of Committee E-2.

It was announced that the committee had completed the preparation of proposed revisions in the Tentative Method of Quantitative Spectrochemical Analysis of High Grade Pig Lead for Copper, Bismuth, Silver and Nickel (E 25 – 35 T) and Method of Quantitative Spectrochemical Analysis of Zinc Alloy Die Castings for Minor Constituents and Impurities (E 27 – 35 T) which, if approved by letter ballot of the committee, would be submitted to the Society through Committee E-10 on Standards subsequent to the annual meeting.

The report of Committee E-3 on Chemical Analysis of Metals was presented by T. A. Wright. In presenting the report Mr. Wright submitted the following resolution for inclusion in the report as finally printed, which subject is to receive consideration in the committee during the year.

> "Methods of Sampling and Analysis devised by Committee E-3 shall show as close an approach to accuracy as is in accord with allowable costs and good analytical practice."

There being no further business the chairman then declared the Fortieth Annual Meeting adjourned *sine die*.

July, 1937.

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[1] This recommendation was submitted to letter ballot of Committee B-7, which consists of 41 members; 33 members returned their ballots, of whom 27 voted affirmatively and 0 negatively.

[2] This recommendation was submitted to letter ballot of Committee B-7, which consists of 41 members; 33 members returned their ballots with the following results: B 26 – 36 T, affirmative 28, negative 0; B 108 – 36 T, affirmative 29, negative 0.

FUELS OF TODAY AND TOMORROW

Annual Address by the President

Arno C. Fieldner[1]

June 28, 1937

Thirty years ago when I entered the service of the Government Fuel Testing Laboratory in Pittsburgh, I had no idea that some day I should have the honor of addressing my colleagues in the materials field on the vitally important subject of our fuel supply and the present-day trends in its utilization.

At that time coal furnished 89 per cent, oil 8 per cent, and natural gas 3 per cent of the mineral fuel energy of the United States; water power contributed an equivalent equal to 3 per cent of the mineral fuels. In 1907, King Coal was the undisputed monarch of the fuel world, and no one anticipated that in thirty years his kingdom would be reduced to one-half of the total energy supply. As shown in Figs. 1 and 2 (Annual Supply of Energy from Mineral Fuels and Water Power in the United States) the decline was gradual. The effect of petroleum and water power on the demand for coal was not noticeable, due to the accelerating industrial activity created by the World War; and the fact that during this period the automobile was stimulating new conceptions of transportation and displacing horses rather than trains and trolley cars, which received their energy from coal.

After the war, the displacing effect, first of petroleum, then of natural gas caused a steady decline in the proportion of energy from coal until 1933, the bottom year of the depression. This decline was augmented by the cumulative effect of research on increasing the efficiency of utilization of coal in many uses.

Rise of Petroleum

The present age of intense competition of the different fuels has its underlying cause in the last century, when Otto invented the gas engine in 1867 and Daimler built the first motor car using gasoline in 1887. Next to the primitive urges of life, man, especially modern man, desires most to move rapidly from place to place. When the automobile gave promise to fill this want, the industry attracted to its ranks not only the adventurous spirits of the day but the keenest engineers in the profession—men who were attracted by the pioneering opportunities afforded by a growing industry. This situation spread to the production of the accessory materials —rubber, alloys, steel, and road materials—and above all gave rise to the great American petroleum industry as we have it today, based primarily on providing the fuels and lubricants for a vast transportation fleet numbering some 28 million motor vehicles, enough to move simultaneously the entire nation and a large proportion of its goods as well.

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[1] Chief, Technologic Branch, U. S. Bureau of Mines, Washington, D. C.

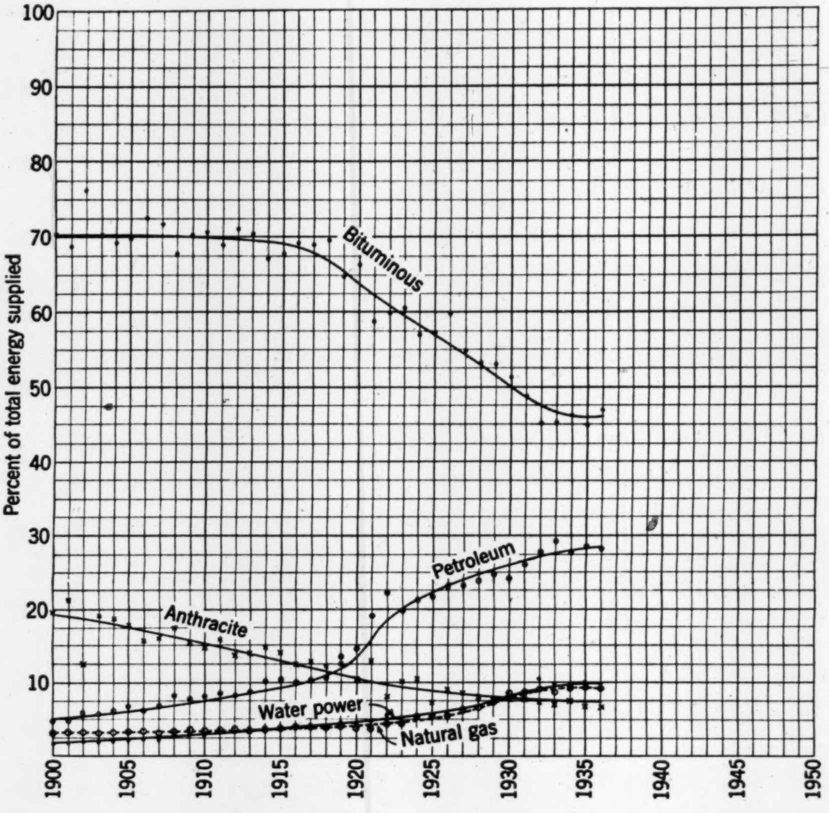

FIG. 1.—Percentage of Total Energy Supplied by Mineral Fuels and Water Power.

Data from Coal Economics Division, U. S. Bureau of Mines. The fuel equivalent of water power is taken in each year at 4 lb. coal per kw-hr., the average, in round numbers, of central-station practice in 1913.

Search for Oil and Improvement of Production.—Thirty years ago, an annual production of 166 million barrels of oil supplied the world with lamp and lubricating oils and was beginning to force its way to the front as a fuel for steam generation in competition with coal. By 1915, automotive needs for gasoline began to be felt. Prospecting spread to many states, and the science of petroleum geology greatly helped in new discoveries. Following the war, demand again overtook supply. Thirty-four thousand wells, drilled in 1920 alone, brought in prolific fields. A flood of oil from California and the mid-continent replenished the deficit.

In 1926 another shortage alarm appeared. The number of motor vehicles had increased from 9 million in 1920 to 22 million in 1926. The domestic gasoline supply was falling behind the demand. New efforts were put forth in various directions, and serious consideration was given to the possibilities of supplemental motor fuels from oil shale, coal, and vegetable materials. However, this fear was shortlived. Deeper drilling—to depths exceeding 7000 ft., then to 10,000 ft., and recently to 12,000 ft.—and most of all, the introduction of geophysical methods in finding oil strata, provided new discoveries at an increasing rate; and in addition the

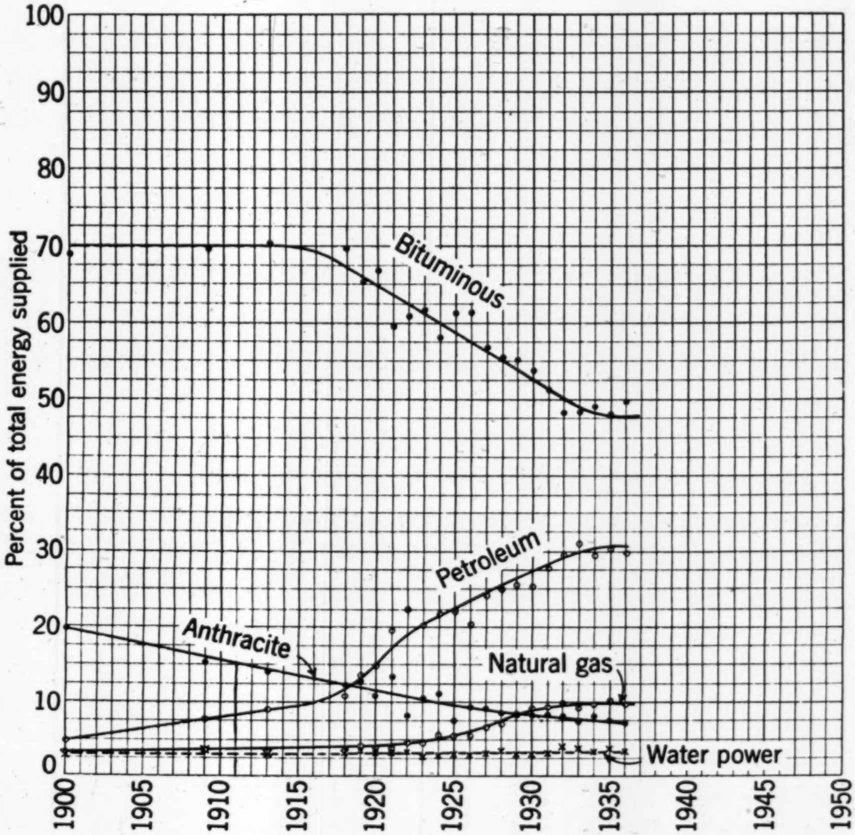

FIG. 2.—Percentage of Total Energy Supplied by Mineral Fuels and Water Power.

Data from Coal Economics Division, U. S. Bureau of Mines. The fuel equivalent of water power is taken at the prevailing average of all central stations for each year, declining from 6.45 lb. per kw-hr. in 1902 to 1.44 lb. in 1936.

enormous East Texas pool with 22,000 wells and an area of some 200 square miles, was discovered in 1930 by wild-cat methods. Slightly over 1 billion barrels of oil were produced in the United States in 1929, and this figure was duplicated in 1936.

Developments in Refining Petroleum.—Parallel with the finding of new sources of crude oil, there was continued progress in increasing the yield and quality of the gasoline produced. Gasoline, formerly a dangerous contaminant of kerosine and fuel oil, became the most valuable petroleum product. Physical separation of this naturally occurring fraction soon became insufficient to supply the need. The good old Pennsylvania crude oils contained 20 to 25 per cent of gasoline, but many of the newly discovered oils contained less than 10 per cent. Science again came to the rescue. As the geologist helped the prospector to find more oil, so the chemist aided the refiner in making more gasoline. By applying heat and pressure the complex molecules of heavy crude oil were broken down to the simpler ones of gasoline. Burton, who accomplished this task commercially in 1912, initiated a series of refining improvements that have increased the production ratio of gasoline to crude oil from 13 per cent in 1912 to 44 per cent in 1935.

The introduction of continuous pipe stills in the early 20's, the use of higher temperatures and pressures permitted by new alloys, and the application of chemical engineering principles in the condensation and fractionation of volatile products have resulted in greatly increased and vastly improved yields. The present average yield of gasoline by cracking is estimated at 60 per cent; and each grade of fuel marketed throughout the broad expanse of the United States has uniform properties. Great credit is due to the Cooperative Fuel Research Committee supported by the petroleum and automotive industries, and to the Committee on Petroleum Products and Lubricants of this Society, for appraising the essential properties of motor fuels and for standardizing the methods for determining these properties. No such nationwide improvement could have been accomplished without their work.

Another landmark in the improvement of motor fuels was Midgley and Boyd's discovery, inspired by Kettering, in 1922, that the addition of small quantities of tetraethyl lead and certain other substances to gasoline would suppress detonation and permit the use of higher compression ratios in the engine, thus increasing the efficiency of utilization. Concurrently with the use of lead came the extension of cracking to kerosine and residual oils and the re-forming of gasoline itself into structures that burned with less tendency to knock. By addition of lead and also by chemical modification, the anti-knock properties of gasoline have been improved from an average octane rating of 59 for regular-grade gasoline in 1930, to 70 in 1936. Premium grade now rates 78 and aviation grades of 100 are beginning to be used.

The average compression ratio of automobile engines sold in 1925 was 4.1 to 1. The general availability of anti-knock motor fuels has permitted an increase in the average ratio to as much as 6.0 to 1 in 1936. So great has been the bromine requirement of the lead anti-knock compounds that it became necessary for chemists to devise a process for extracting the minute quantities of bromine in sea water. A plant was built on the coast of North Carolina which now is extracting about 46,000 lb. of bromine per day from 145 million gallons of sea water.[1]

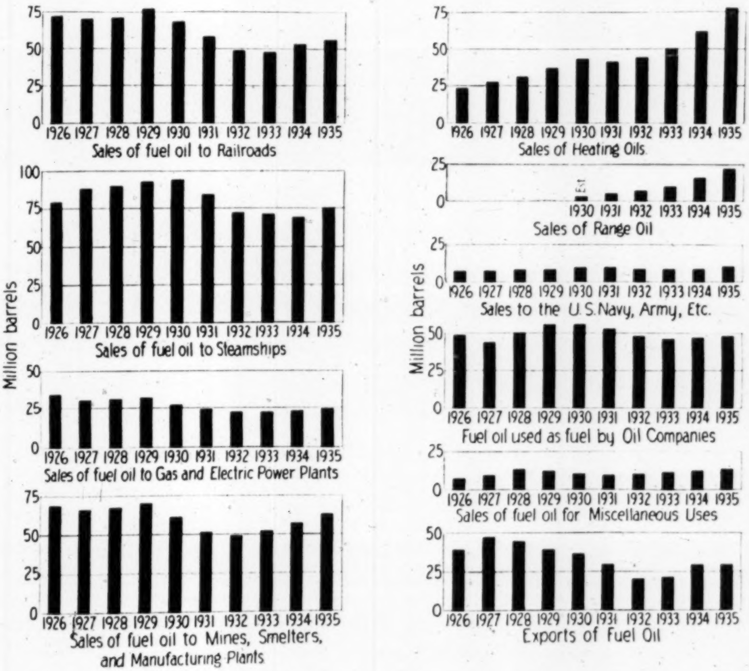

FIG. 3.—Sales of Fuel Oils and Range Oil by Uses, 1926 to 1935.
Graph by Petroleum Economics Division, U. S. Bureau of Mines.

These major developments in the production of motor fuel, together with a number of minor advances, have added some 30 to 50 per cent to the effectiveness of our supply from crude petroleum. From 1920 to the end of 1936, cracking alone is estimated to have produced an amount of gasoline which without cracking would have required the production of an additional 8½ billion barrels of crude oil.[2]

Displacement of Coal by Fuel Oil.—Since the earliest days of the petroleum industry, refining operations have been adjusted to the market demands of the lighter fractions. Before 1910, kerosine and afterward gasoline set the pace. The residual portions, almost half of the total crude, sought and found a receptive market as gas oil and fuel oil. Like any other by-product, the price of these oils was set by prevailing fuels.

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[1] *Industrial and Engineering Chemistry,* News Edition, Vol. 15, March 10, 1937, p. 105.

[2] Fred van Covern, "Status and Outlook of Petroleum Supply, Demand and Stocks," *American Petroleum Institute Quarterly,* Vol. 7, April, 1937, p. 9.

Liquid fuel was tried on ships before the World War, and by its close virtually all the navies were burning oil. The merchant marine followed the naval example; and the oil-fueled tonnage of the world rose from 18 per cent in 1920 to 40 per cent in 1930 and to 50 per cent in 1936. Forty per cent of the oil-fueled tonnage of 1936 consisted of Diesel-powered ships.

The railroads also have been, and are today, large consumers of fuel oil. While this use has been limited largely to the Southwestern and Western states, where coal is less available and fuel oil abundant, the recent advent of Diesel-motored trains is encroaching on the use of coal in other regions.

TABLE I.—SALES OF GAS OIL AND FUEL OIL,[a] 1930–35, BY USES.

Thousands of barrels of 42 gal.
Data furnished by Petroleum Economics Division, U. S. Bureau of Mines.

Uses	1930	1931	1932[b]	1933[b]	1934	1935
Railroads	67 900	58 150	48 908	48 305	52 581	55 651
Steamships (including tankers)	94 152	83 559	72 531	70 445	69 262	74 581
Gas and electric power plants	26 769	24 490	22 199	22 507	23 143	23 647
Smelters and mines[c]	6 841	4 363	2 130	2 538	2 682	2 448
Manufacturing industries[c]	53 921	46 873	46 370	48 962	54 260	61 128
Heating oils	43 279	40 578	44 264	50 140	60 822	76 853
U. S. Navy, Army transports, etc	8 681	9 203	7 968	8 000	7 914	10 428
Oil-company fuel	55 943	52 710	47 700	46 200	47 404	48 116
Miscellaneous uses	9 875	9 211	9 500	11 250	12 253	13 133
Total United States	367 361	329 137	301 570	308 347	330 321	365 985
Exports and shipments	36 450	29 231	19 994	20 563	28 605	28 948
	403 811	358 368	321 564	328 910	358 926	394 933
Range oil	3 000[d]	4 549	6 841	10 269	15 756	21 526

[a] Includes some crude oil burned as fuel.
[b] Partly estimated.
[c] Revised figures for 1932, 1933, and 1934.
[d] Estimated.

Since 1923, the use of fuel oil has spread to various industries, especially for operations requiring flexibility of heating, accurate temperature control, and freedom from ash. The changing distribution of fuel-oil consumption since 1926 is shown in Fig. 3 and Table I. The most important recent trend is the ever-accelerating gain of oil-heating for homes, apartments, and office buildings. The sale of heating and range oils increased from 25 million barrels in 1926 to almost 100 million barrels in 1936. One hundred million barrels of oil is equivalent to 24 million tons or one-half of the present production of anthracite. In all its uses the consumption of fuel oil alone is equivalent to about 100 million tons of coal—slightly more than one-fifth of the total production of anthracite and bituminous coal.

RISE OF NATURAL GAS

Next to petroleum, the most striking development of our bountiful fuel supply is the utilization of natural gas. Once the source of eternal flames of ancient worshipers of fire, it is now a premier fuel supplied through thousands of miles of pipe to heat homes and cook meals in half the area of the United States. In the producing fields it generates power for pumping gas and oil, it furnishes carbon black for filler in automotive tires, and it is becoming an important raw material for new chemical products. A few deposits contain the non-flammable helium, now indispensable for airships.

Although used for lighting Fredonia, N. Y., as early as 1821, the use of natural gas was sporadic until the discovery of reservoirs of gas in the search for oil. The first iron pipe line for natural gas—a 2-in. line 5½ miles long—was built in 1872 from the Newton well to Titusville, Pa.

Other wells and new discoveries followed, first in Pennsylvania, then in West Virginia, Ohio, Kentucky, Indiana, the mid-continent and California. The natural gas of the Appalachian field was more quickly absorbed by the centralized industries and homes of the district than it was in the sparsely industrialized sections of the Southwest, where later extensive reservoirs of oil and gas in Oklahoma, Louisiana, Texas and California were opened. The original waste of gas in these fields was enormous, but is declining with the lengthening of pipe lines to industrial centers. The Munroe field of Louisiana, discovered in 1916, subsequently indicated a reserve of 5 trillion cubic feet of gas. This was enough to justify long-distance pipe lines to centers of consumption; beginning in 1926, pipe lines were built successively to New Orleans, Memphis, St. Louis, Birmingham and Atlanta. The largest reserve of all proved to be the Amarillo, or Panhandle field in Texas. Estimates of the original content vary from 14 to 20, and even as high as 41 trillion cubic feet of gas.[1] Pipe lines from this field extended to Ft. Worth, Denver, Kansas City, and eventually to Chicago, Minneapolis and Detroit. This development was made possible through metallurgical research in making steel and fabricating it by electric or acetylene gas welding into virtually continuous pipes capable of transporting gas at working pressures of 600 lb. per sq. in. and more, with very small leakage over a distance of 1000 miles. Enormous quantities of natural gas, a most valuable form of fuel heretofore lacking a market, were made available for domestic and commercial use.

Natural Gasoline and Bottled Gas.—Fifty-five per cent of the natural gas produced and consumed in the United States comes to the surface of the ground in association with oil. Even the so-called "dry" gases contain appreciable quantities of propane, butane, pentane, and higher members of the paraffin series of hydrocarbons. From 0.5 to 10.0 gal. of gasoline and condensable hydrocarbons can be recovered from 1000 cu. ft. of "wet" gas by compression and refrigeration or by absorption in oil.

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[1] Victor Cotner and H. E. Crum, "Geology and Occurrence of Natural Gas in Amarillo District, Texas," *Bulletin*, Am. Assn. Petroleum Geologists, Vol. 17, No. 8, August, 1933, p. 877.

W. K. Cadman, "Kansas Natural Gas" (reports estimate (p. 24) of Panhandle Field gas reserve made by R. C. Moore, State Geologist of Kansas), Report to the Kansas State Planning Board, December, 1934.

By 1913, the growing demand for gasoline had led to the installation of 250 plants east of the Mississippi for the recovery of this so-called "natural gasoline." The industry proved profitable and grew steadily, mounting to a production of 9.2 million gallons of natural gasoline as early as 1920. This amount was 7 per cent of the total gasoline production, and the average yield was about 0.8 gal. per 1000 cu. ft. of gas treated.

In the early days of the industry, the volatile propane, butane, and pentane fractions were allowed to boil off in the atmosphere to obtain a stable product suitable for blending with refinery gasoline. This procedure not only wasted these volatile fractions, but considerable proportions of stable gasoline were lost. Beginning about 1924, the application of modern chemical engineering in the use of bubble-type absorbers and stills with efficient rectifying columns eliminated these losses. The volatile fractions were recovered and transported under pressure in steel bottles for domestic and industrial use.

Thus the application of scientific research eliminated another serious waste of resources and provided a new fuel known as bottled or liquefied gas. This fuel combines the convenient and accurate control of a gas with the portability and flexibility of gasoline. Beginning at the modest figure of 223,000 gal. in 1922, the marketed production of liquefied petroleum gases increased literally by leaps and bounds to a total production of 107 million gallons in 1936.

Twenty-eight per cent of the bottled gas, in the form of propane, went to domestic users who live beyond the reach of gas distribution systems; 60 per cent, in the form of the less-volatile butane and propane-butane mixtures, was sold chiefly as industrial fuel and to gas companies for distribution through gas mains in small communities.

Assuming a calorific value of 98,000 B.t.u. per gallon for the bottled gas sold in 1936, it amounts to as much as 10 per cent of the total energy of the manufactured gas (223 billion cu. ft.) sold to domestic consumers in 1935. The industry is expected to grow to much greater proportions, as much larger quantities are available both from natural gas and from the still gases of petroleum refineries.

Polymerization of Hydrocarbons.—The most recent new developments in the improved utilization of hydrocarbons are the polymerization processes for the production of high anti-knock gasoline.[1]

Here the chemist has reversed the cracking process of breaking down large molecules of heavy oil into the smaller gasoline molecules. Polymerization consists of building up molecules of gasoline size by uniting gaseous and light-vapor molecules, such as the unsaturates, ethylene, propylene,

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[1] Minerals Yearbook, U. S. Bureau of Mines, pp. 647–648 (1936).

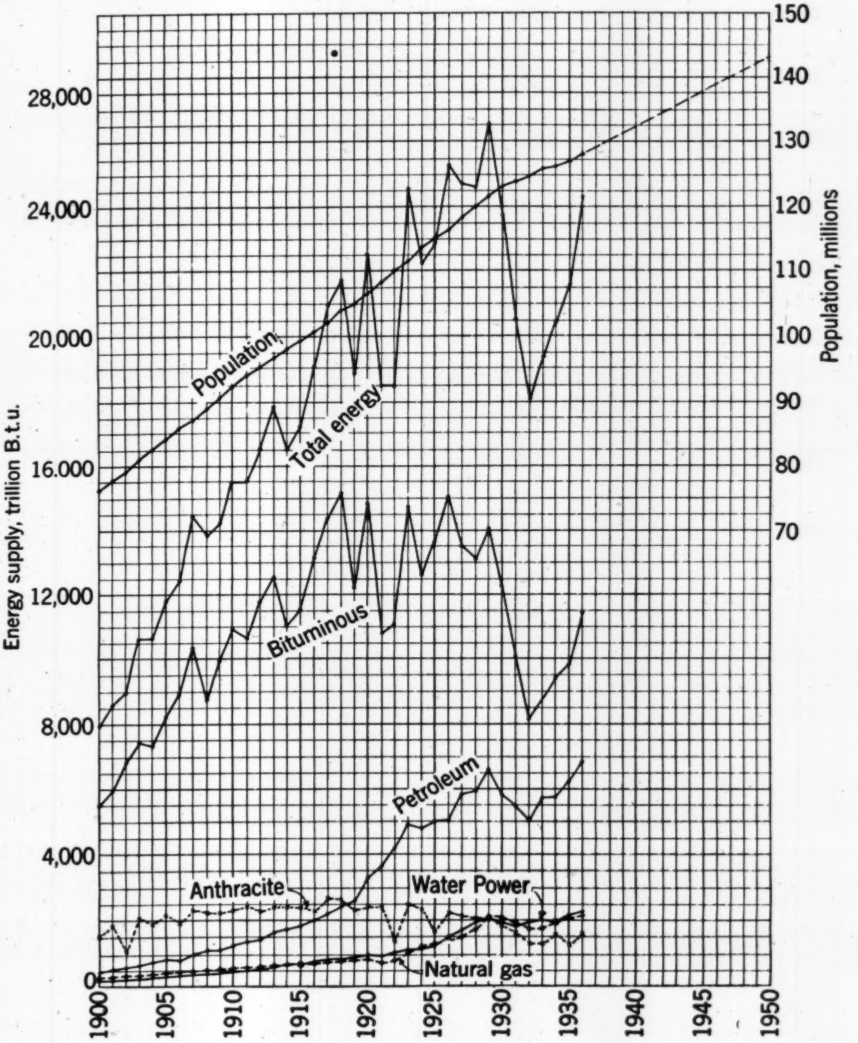

FIG. 4.—Annual Energy Supply of the United States.

Data from Coal Economics Division, U. S. Bureau of Mines. The fuel equivalent of water power is taken in each year at 4 lb. coal per kw-hr., the average of central-station practice in 1913.

and butylene, present in large proportion in cracking-still gases, and the saturated hydrocarbons ethane, propane and butane found in natural gas.

Two types of commercial polymerization processes began operation in 1935. These types are known as the "catalytic" and the "thermal" processes.[1] In the "catalytic gas polymerization process," olefin-containing gases from cracking stills or from the catalytic dehydrogenation or cracking of propane and butane are heated to a temperature of about 230 C., at a pressure of 200 lb. per sq. in. in the presence of a phosphoric acid catalyst; in the "thermal processes," mixtures of gaseous paraffin and olefin hydrocarbons are treated at temperatures ranging from 480 to 700 C., and 500

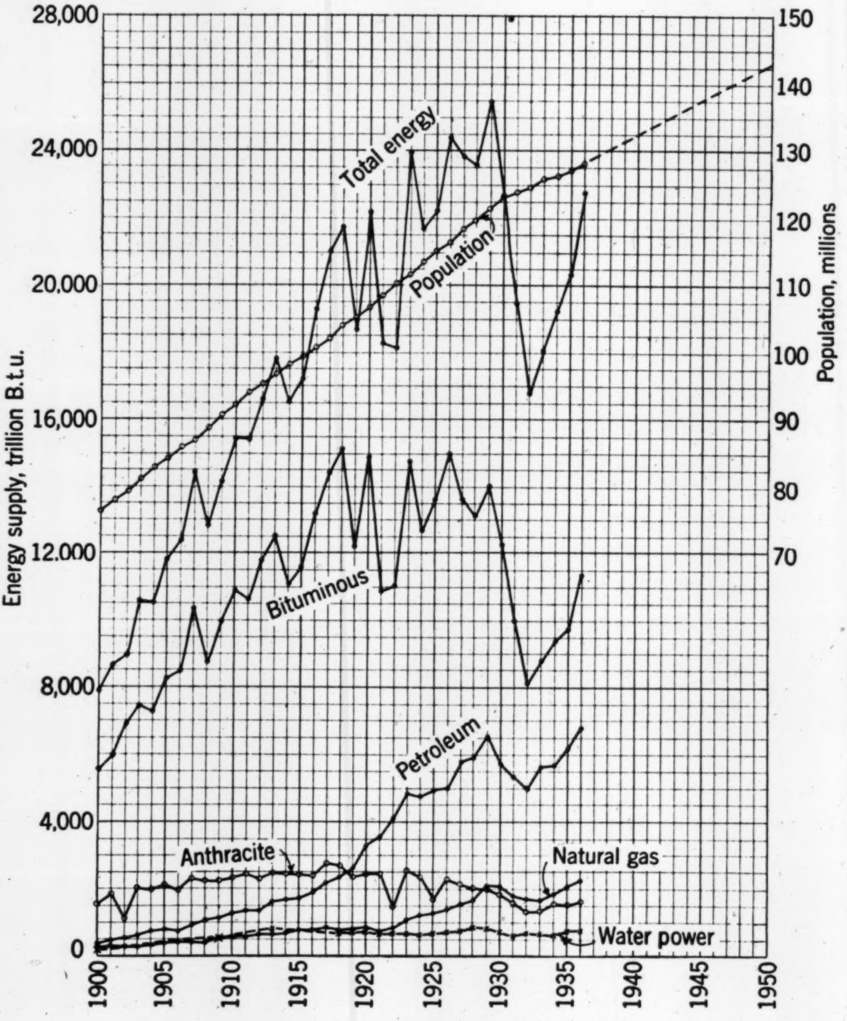

FIG. 5.—Annual Energy Supply of the United States.

Data from Coal Economics Division, U. S. Bureau of Mines. The fuel equivalent of water power is taken at the prevailing average of all central stations for each year, declining from 6.5 lb. per kw-hr, in 1902 to 1.44 lb. in 1936.

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[1] G. Egloff, "Polymer and Isoöctane Motor Fuels," *Industrial and Engineering Chemistry*, News Edition, Vol. 15, pp. 245–246 (1937).

to 3000 lb. per sq. in. pressure. The gasoline obtained by these processes has octane numbers ranging from 76 to 86 and therefore has a premium value for blending with ordinary gasoline. About 6 gal. of gasoline are reported to be obtained by the catalytic process from 1000 cu. ft. of a refinery gas containing 37 per cent of propylene plus butylene.

The combined annual capacity of polymerization plants, in operation, under construction and proposed at the end of 1936, was estimated at 5 to 10 million barrels.[1] Completion of a pipe line from the East Texas field to a polymerization plant on the Gulf Coast indicates that production of this type of anti-knock gasoline will continue to enlarge, and that an unlimited market for butane is assured in the production of high octane gasoline for aviation or blending with ordinary gasoline. Estimates of potential production from refinery and natural gas range from 10 to 25 per cent (and higher[1]) of the 1936 gasoline demand of 482 million barrels.[2,3]

Another probability is the direct use of butane as a motor fuel. Compression ratios of 10 to 1 are possible without detonation. Combustion is complete, and higher efficiencies are obtainable than with gasoline. The use of propane and butane as fuel for stationary units is increasing, and interesting experiments have been made in combining power production with refrigeration on trucks and with air cooling of busses.[4]

COAL PRODUCTION AND UTILIZATION

Let us return now to the changing use of coal—once our only mineral fuel—now supplying but half of the total energy demand. From 1898 to 1918 the production of bituminous coal increased at an average rate of approximately 20 million tons per annum, reaching a maximum of 579 million tons or 15 180 trillion B.t.u. in 1918 (Figs. 4 and 5). That was the end of the rise. From that date the demand for bituminous coal wavered with the ups and downs of industry until in 1927 began a decline which ended at 310 million tons in 1932 (8114 trillion B.t.u.).

In 1936 the production had returned to 434 million tons (11,373 trillion B.t.u.). Thus, the coal industry, geared to an annual increase of 20 million tons per annum, was faced abruptly at the end of the war with an increasing capacity and a declining demand. Profits vanished, and whole communities were deprived of work. Many mines were closed, and intense competition ensued. The inroads of competitive fuels began to be realized, and measures were taken to hold the markets for coal. These consisted of lowering the costs of production and improving the quality of the product.

Improved machinery was designed for cutting coal at the face and loading it mechanically into cars. Underground conveyors were installed—

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[1] G. Egloff, "Polymer and Isoöctane Motor Fuels," *Industrial and Engineering Chemistry*, News Edition, Vol. 15, pp. 245–246 (1937).

[2] Arch L. Foster, "Conservation of Oil Given New Properties by Polymerization Process," *National Petroleum News*, Vol. 27, No. 47, November 20, 1935, p. 73.

[3] L. C. Snider and B. T. Brooks, "Probable Petroleum Shortage in the United States and Methods for Its Alleviations," *Bulletin*, Am. Assn. Petroleum Geologists, Vol. 20, p. 46 (1936).

[4] G. G. Oberfell, "Progress of the Liquefied Gas Industry," *Gas Age Record and Natural Gas*, February 18, 1937, pp. 29–31, 42.

in one instance 4 miles long. Twelve per cent of the 1935 production was mechanically mined, and mechanization is continuing at a faster pace.

Reduction in the cost of mining has been paralleled by improvement of the quality of coal. Washeries and cleaning plants have been installed at many mines for reducing ash and sulfur to the lowest practicable limits. About 15 per cent of the 1935 production was washed or air-cleaned. The best preparation plants provide fuel of definite size gradation and constant ash and sulfur content.

Residential Stokers.—To hold the house-heating trade, coal producers are cooperating with appliance manufacturers in the development of automatic stokers, which provide much of the convenience of gas or oil-fired furnaces at a lower cost for fuel. The sales of domestic stokers have increased from 7000 in 1932 to 76,000 in 1936. The dust nuisance in handling coal has been abated by spraying coal with oil or hygroscopic solutions, and even packaged fuel consisting of briquets wrapped in kraft paper has been introduced for disposing of the dust from high-grade coals that accumulates in distributing yards.

Increase of Fuel Efficiency.—In the generation of power from coal, the public utilities and the power-plant-equipment manufacturers have made great strides in improving fuel efficiency. From an average consumption of 7.05 lb. of coal per kilowatt-hour in 1899 and 3.2 lb. in 1919 to 1.44 lb. in 1936 is a truly remarkable record. Since the most efficient modern stations are producing 1 kw-hr. from 0.8 lb. of coal, further decrease of the average may be expected as new equipment replaces the old.

The higher efficiencies obtained with the introduction of pulverized coal in the period following the war have now been equaled by stoker-fired installations. Both types of boiler plants give steam efficiencies of 90 to 92 per cent. The slag-tap furnace, with powdered-coal combustion, avoids clinker trouble. Coal of low ash-fusing temperature may be used and the ash removed in molten form. This advantage of pulverized-coal combustion has been met in stoker practice by preventing fusion of ash through water-cooling the tuyères of underfeed stokers. This device permits the use of coals having a low ash-softening temperature and a high ash content. The spreader-type stoker[1] now coming into use is adapted to coals of widely varying properties. It is an example of the trend toward equipment suitable for any kind of coal.

The best boiler furnaces are nearing their maximum possible efficiency. Further improvement of total fuel-power efficiency must be obtained through the conversion of steam to power. Metallurgical progress in the development of high-temperature alloy steels permits the use of higher steam pressures. The best record for a coal-steam-turbine plant is

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[1] J. F. Barkley, "The Forced-Draft Spreader Stoker," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 59, pp. 259–265 (1937).

10,900 B.t.u. per kw-hr., or 31 per cent efficiency. This was obtained at the Port Washington power plant[1] at Milwaukee, Wis., operating at 1230 lb. per sq. in. pressure and at a temperature of 825 F. Mercury-vapor plants, still in the developmental stage, have given 32 per cent efficiency in regular operation and on test might go as high as 40 per cent.[2]

Carbonization and Gasification of Coal.—As the modern central power station represents our greatest advance in the combustion of coal, so does the modern by-product coke-oven represent our major development in the processing of coal. Thirty years ago, 85 per cent of our metallurgical coke was made in beehive ovens without recovery of gas and by-products. This waste has stopped. Less than 5 per cent of the coke produced today comes from beehive ovens. By-product oven installations increased very slowly until 1914. Then the World War created an acute demand for the by-products benzol and toluol, as well as metallurgical coke. American industry met the demand, and an active period of construction ensued. Designs were improved, and following the war, the industry entered the field of city gas supply. This move was accompanied by a vigorous educational campaign on the domestic use of coke. Beginning at 2,300,000 tons in 1921 sales of domestic coke climbed steadily to 10,500,000 tons in 1934—an amount equivalent to the fuel oil sold for similar purposes during the same year.

Fifteen years ago, the prospects of the coal-carbonization industry seemed very bright. The public was learning to burn coke and to appreciate its smokeless properties; the demand for gas was increasing, and the supply of natural gas from the Appalachian field was declining; the demand for gasoline was mounting rapidly, and discovery of new fields seemed inadequate. Further processing of coal for coke, gas, and liquid by-products appeared inevitable; but unfortunately for the coal industry, the situation changed. A prolific period of discovery was followed by a veritable flood of oil and a hurricane of gas. Natural gas piped from the remote interior to cities of the Great Lakes and virtually to the Atlantic Seaboard stopped further extension of manufacturing gases from coal. Other factors also depressed the economics of coal carbonization; at present, metallurgical coke is the only product from the processing of coal that does not have its price realization fixed by a competitive product: light oil is controlled by gasoline; tar by fuel oil; ammonium sulfate by synthetic ammonia; and manufactured gas by natural gas. The return of normal business activity will bring some coke-oven construction needed to supply a larger steel production and possibly a larger demand for solid smokeless fuel, but no extensive program of oven building need be expected until natural gas declines again.

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[1] A. G. Christie, "Twenty-five Years of Power Development," *Combustion*, April, 1937, pp. 34–35.

[2] A. G. Christie, "Development and Performance of American Power Plants," *Mechanical Engineering*, Vol. 58, September, 1936, pp. 539–561.

Low-Temperature Carbonization.—The commercial development of low-temperature carbonization, which provides an easily ignitable smokeless fuel, likewise has been retarded in the United States by the lack of demand for the light and heavy oils that are yielded in much greater volume than in the high-temperature process.

Despite the spectacular failures of two large commercial plants technical progress has been made, and a limited production has been marketed during the past three years.

Complete Gasification.—Producer gas from coal or coke, once an important fuel for heating furnaces, has declined steadily in use. Surplus gas and tar from by-product coke-ovens, natural gas, and fuel oil have taken its place. Carburetted water-gas equipment continues to be an important adjunct to city gas plants to carry peak loads or interruption of supply. New modifications involving the re-forming of natural gas or refinery gases have been developed.

The increasing availability of gas from various sources has changed the emphasis in city gas utilities from manufacturing gas to mixing purchased gases and distributing them to consumers. The proportion of purchased gas of the total of purchased and manufactured gas increased from 29 per cent in 1929 to 35 per cent in 1934. These figures do not include gas distributed to the ultimate consumers by the natural-gas industry. A direct comparison of these two groups shows that for the year 1936 natural gas comprised 56 per cent of the volume of gas and 68 per cent of the energy supplied in gaseous form to domestic and commercial consumers. Since the total annual production of natural gas is four to five times the amount of natural gas distributed to domestic and commercial consumers by public-utility gas companies, it is evident that further increases in the ratio of natural to manufactured gas may be expected for several years to come.

PRESENT-DAY FUEL SUPPLY AND DEMAND

Having reviewed the changing conditions of fuel supply and demand during the last 30 years, let us consider now our present situation. We have solid, liquid, and gaseous fuels in abundance, and we are using them at an increasing rate. The total energy supply from these mineral fuels, in trillions of B.t.u.'s, increased from 14,000 in 1907 to 24,600 in 1929—a 75 per cent increase in 22 years. The depression caused a drop to 16,122, but recovery had raised the figure to 21,878 by 1936. The changing proportions of this energy contributed by the several mineral fuels and by water power was shown in Figs. 1 and 2.

The distribution of energy consumption in 1929, a year of high industrial activity, is given in Tables II and III.

In round numbers, industry and public utility power consumed 45

TABLE II.—DISTRIBUTION OF ENERGY CONSUMPTION OF MINERAL FUELS IN 1929.

	Coal, million net tons[a]			Petroleum, million barrels[b]					Natural Gas[f]	
	Anthracite	Bituminous		Gasoline[c]		Kerosine[d]	Fuel and Gas Oil[e]			
	Tons	Tons	Per cent	Barrels	Per cent	Barrels	Barrels	Per cent	Billion cu. ft.	Per cent
Transportation	....	143	27.7	351	92.9	....	174	47	....	...
Railroads	....	135[g]	26.2	1.1	0.3	....	76	20.5	....	...
Ships and boats	....	8	1.5	0.8	0.2	....	98	26.5	....	...
Road vehicles and airplanes	....	....	....	349.1	92.4	....	....	....	....	...
Public-utility power	....	38	7.3	....	....	....	10	2.7	113	6
Public-utility gas	....	6	1.3	....	....	....	22	6.0	....	...
Industrial	....	225	43.3	13.6	3.6	....	127	34.3	1444[h]	75
Domestic and miscellaneous[i]	69	106	20.4	13.2[j]	3.5	36	37	10.0	360	19
Totals	69	518	100	378	100	36	370	100	1917	100

[a] Mineral Resources of the United States for 1929, U. S. Bureau of Mines, p. 771.

[b] 135 million barrels of petroleum consumed in the production of refinery gas, coke, lubricants, wax, road materials, etc., are not included in this distribution.

[c] Data supplied by Economics Branch, U. S. Bureau of Mines.

[d] G. R. Hopkins, "Petroleum Refinery Statistics, 1929," *Bulletin 339*, U. S. Bureau of Mines, p. 5 (1931).

[e] *Ibid.*, p. 89.

[f] Mineral Resources of the United States for 1929, U. S. Bureau of Mines, p. 320.

[g] Includes 13 million tons of coal used in shops and stations.

[h] Two-thirds of this amount used for fuel in gas and petroleum field operations, and fuel in oil refineries, and for the production of carbon black.

[i] Includes minor industrial uses.

[j] Includes agricultural uses.

TABLE III.—DISTRIBUTION OF ENERGY CONSUMPTION FROM MINERAL FUELS IN 1929 IN TRILLIONS OF B.T.U.[a]

	Coal		Petroleum			Natural Gas	Totals	Per cent of Total Energy Consumption
	Anthracite	Bituminous	Gasoline	Kerosine	Fuel Oil			
Transportation	....	3 720	1790	...	1027	....	6 537	31
Railroads	....	3 512	6	...	448	....	3 966	19
Marine	....	208	4	...	579	....	791	4
Road vehicles and airplanes	....	.....	1780	...	....	....	1 780	8
Public-utility power	....	990	....	...	59	113	1 162	5
Public-utility gas	....	156	....	...	130	....	286	1
Industrial	....	5 850	70	...	749	1444[b]	8 113	38
Domestic and miscellaneous[c]	1794	2 760	67	198	218	360	5 397	25
Totals	1794	13 476	1927	198	2183	1917	21 495	100
Per cent of total energy, all fuels	8	63	9	1	10	9		100

[a] Heating values used in conversion:
1 ton coal = 26 million B.t.u.
1 bbl. gasoline = 5.1 million B.t.u.
1 bbl. kerosine = 5.5 million B.t.u.
1 bbl. fuel oil = 5.9 million B.t.u.
1000 cu. ft. natural gas = 1 million B.t.u.

[b] Two-thirds of this amount used for carbon-black production, and for fuel in gas and petroleum field operations, and for oil-refinery fuel.

[c] Includes minor industrial uses.

TABLE IV.—PERCENTAGE OF ENERGY SUPPLIED BY THE SEVERAL MINERAL FUELS FOR CERTAIN MAJOR USES IN 1929.

Kind of Fuel	Domestic and Miscellaneous	Public-Utility Power, Gas and Industrial	Railroads	Ships	Road Vehicles and Airplanes	Total Transportation
Coal	84	73	89	27	...	57
Petroleum	9	11	11	73	100	43
Natural gas	7	16[a]	...	...	...	...
Totals	100	100	100	100	100	100

[a] Two-thirds of this 16 per cent used for gas and petroleum field operations, oil-refining fuel, and production of carbon black.

per cent, transportation about a third, and domestic and miscellaneous uses one-fourth of our energy supply. Coal constituted (see Table IV) 84 per cent of the fuel for domestic and miscellaneous purposes, 73 per cent for industrial use and public-utility power, and 57 per cent for transportation; petroleum provided 43 per cent of transportation fuel, including almost 100 per cent of road vehicles and airplane fuel, 73 per cent of marine fuel, and 11 per cent of railroad fuel. Natural gas comprised 16 per cent of industrial and public-utility fuel consumption and 7 per cent of the domestic and miscellaneous needs. Since 1929, 7 per cent of the total energy demand has been transferred from anthracite and bituminous coal to petroleum, natural gas, and water power (see Tables V and VI). The continuation of this shift concerns not only the operators and workers in the coal and oil industries, but the nation itself with respect to future needs.

TABLE V.—ANNUAL SUPPLY OF ENERGY FROM MINERAL FUELS AND WATER POWER IN THE UNITED STATES,[a] TRILLIONS OF B.T.U.

	1899	1913	1923	1929	1936
Water power at constant fuel equivalent:[b]					
Anthracite	1643	2 490	2 539	2 008	1 489
Bituminous coal	5065	12 535	14 791	14 017	11 373
Domestic petroleum	342	1 491	4 394	6 044	6 591
Imported petroleum	0	102	492	474	194
Natural gas	240	626	1 082	2 062	2 231
Water power	135	588	1 136	1 929	2 256
Total	7425	17 832	24 434	26 534	24 134
Water power at prevailing fuel equivalent[c]	238	609	680	810	798
TOTAL	7513	17 832	23 978	25 415	22 676

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[a] F. G. Tryon, W. H. Young, L. Mann and J. R. Bradley, "Coal," Minerals Yearbook, U. S. Bureau of Mines, p. 564 (1936).

[b] Equivalent of water power for fuel is taken in each year at 4 lb. coal per kw-hr., the average of central-station practice in 1913. The conversions for other fuels are:

Anthracite, 13,600 B.t.u. per lb.
Bituminous coal, 13,100 B.t.u. per lb.
Petroleum, 6,000,000 B.t.u. per bbl.
Natural gas, 1075 B.t.u. per cubic ft.

[c] Water power converted to fuel equivalent at the average prevailing efficiency of central stations for the year in question, declining from 6.45 lb. per kw-hr. in 1902 to 1.44 lb. in 1936. The following equivalents were used:

1899—7.05 lb. coal per kw-hr.
1913—4.14 lb. coal per kw-hr.
1923—2.41 lb. coal per kw-hr.
1929—1.69 lb. coal per kw-hr.
1936—1.44 lb. coal per kw-hr.

TABLE VI.—PERCENTAGE OF TOTAL B.T.U.'S CONTRIBUTED BY THE SEVERAL MINERAL FUELS AND WATER POWER IN THE UNITED STATES.[a]

	1899	1913	1923	1929	1936
Water power at constant fuel equivalent of 4 lb. per kw-hr.:					
Anthracite	22.1	14.0	10.4	7.6	6.2
Bituminous coal	68.2	70.3	60.5	52.8	47.1
Domestic petroleum	4.6	8.3	18.0	22.8	27.3
Imported petroleum	0.0	0.6	2.0	1.8	0.8
Natural gas	3.3	3.5	4.5	7.7	9.3
Water power	1.8	3.3	4.6	7.3	9.3
Total	100.0	100.0	100.0	100.0	100.0
Water power at prevailing fuel equivalent:					
Anthracite	21.8	13.9	10.6	7.9	6.6
Bituminous coal	67.3	70.3	61.6	55.1	50.1
Domestic petroleum	4.5	8.3	18.3	23.8	29.0
Imported petroleum	0.0	0.6	2.1	1.9	0.9
Natural gas	3.2	3.5	4.5	8.1	9.8
Water power	3.2	3.4	2.9	3.2	3.6
Total	100.0	100.0	100.0	100.0	100.0

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[a] F. G. Tryon, W. H. Young, L. Mann, and J. R. Bradley, "Coal," Minerals Yearbook, U. S. Bureau of Mines, p. 566 (1936).

THE NATIONAL FUEL RESERVES

During 1936 we produced and consumed 31 to 33 per cent of the world production of coal, 60 to 70 per cent of the petroleum, and 95 per cent of the natural gas. We have a little over one-half (3.2 trillion tons of coal and lignite) of the world reserves of coal, and probably 60 per cent[1] of the proved oil reserves. Two-thirds of the oil produced in all countries since the discovery well was drilled in 1859 has come from American wells.

Unlike coal, estimates of the total national reserves of petroleum cannot be made. We can deal only with the proved reserves. In 1934, the United States Geological Survey estimated the oil in the ground recoverable by the current methods of production at 13 billion barrels, and the most recent estimate—that of January 1, 1937, by a committee of the American Petroleum Institute—puts the reserve at virtually the same figure. Thus, in the last three years discovery of new fields has kept pace with production. We cannot forecast future rates of discovery, and opinions differ as to when a decline will begin. Regardless of whether it may begin within one or several decades, it is evident that our oil and gas reserves are small compared to coal.

The deposits of oil shale,[2] largely in the Rocky Mountain States, are estimated to contain a potential supply of 92 billion barrels of crude oil—an amount sufficient to maintain the present annual rate of oil production for nearly 100 years.

The ultimate reserves of natural gas cannot be estimated. Fifty-five per cent of the natural gas is produced in association with oil; therefore, oil exhaustion means the end of natural gas from these particular areas. Reliable estimates of reserves of individual gas fields are being made, but this work has not covered the country as a whole.

Conservative estimates of the proved reserves by individual authorities range from 30 to 40 trillion cubic feet. Other estimates are placed at considerably higher figures. At the present rate of 2 trillion cubic feet of production per year, this amount would last 15 to 20 years. Here also, new sources will be discovered and the life of the fields, no doubt, will be extended greatly as growing social control will prohibit waste and low-grade use of this ideal form of our national fuel supply.

The original reserves of coal and lignite are estimated at 3.2 trillion tons—enough to cover the entire State of Ohio (area, 41,000 square miles) to a depth of 76 ft. Not more than 2 per cent of the original supply of bituminous coal and about 25 per cent of our anthracite has been exhausted. At the 1929 rate of energy consumption, assuming that coal will carry the load after oil, gas, and oil shale are exhausted, and allowing 30 per cent for loss, coal would last 2100 years. (See Tables VII and VIII.)

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[1] V. R. Garfias and R. V. Whetsel, "Proven Oil Reserves: Petroleum Development and Technology," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 118, p. 212 (1936).

[2] Dean E. Winchester, "The Oil Possibilities of the Oil Shales of the United States," *Report II*, Federal Oil Conservation Board, January, 1928, pp. 13–14.

However, there certainly will be an increase in energy demand; an increase at the rate of increase prevailing during the twenties would cut the period to some 500 years, and shortage of supply would be felt in the Appalachian field in 100 years. It must be remembered that the superior coals of the Fastern region comprise only one-seventh of the total reserves. One-half of the total reserve consists of subbituminous coal and lignite. These are inferior fuels, and they are in the western part of the United

TABLE VII.—ESTIMATED MINERAL FUEL RESOURCES OF THE UNITED STATES.

Description	Original Reserve	Reserve End of 1935	Per cent Exhaustion	1929 Production
Anthracite, billions of net tons[a]	22	16.5	25	0.074
Low-volatile bituminous coal, billions of net tons[b]	56	......	4	......
High-volatile bituminous coal, billions of net tons[b]	1378	......	1.6	......
Subbituminous coal, billions of net tons	818	......	0.05	......
Lignite, billions of net tons	940	......	0.01	......
Total, all ranks of coal, billions of net tons	3215	3185	0.9	0.609
Petroleum, billions of bbl.[c]	31 ?	13 ?	58 ?	1.007[d]
Oil in oil shale, billions of bbl.[e]	92	92	0	0
Natural gas, trillions of cu. ft.[f]	80[g] ?	40 ?	50 ?	2.5[h]

[a] M. R. Campbell, "Coal Resources of the United States," U. S. Geological Survey Mimeographed Report (1928).

[b] Includes some medium volatile coal.

[c] W. L. Baker, "U. S. Crude Reserves Enlarged During 1935," *Oil Weekly*, Vol. 80, February 10, 1936, pp. 19–22. Proved reserves based on the amounts recoverable with present methods of production.

[d] 79 million bbl. of crude petroleum were imported; 26 million bbl. of crude petroleum and 123 bbl. of refined products were exported in 1929.

[e] D. E. Winchester, "The Oil Possibilities of the Oil Shales of the United States," *Report II*, Federal Oil Conservation Board, January, 1928, pp. 13–14.

[f] V. R. Garfias, *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 114, pp. 243–244 (1935).

[g] Includes 8 trillion cu. ft. blown into the air and wasted, *Petroleum Times*, Vol. 33, p. 346 (1935).

[h] Includes one-half trillion cu. ft. blown into the air and wasted.

TABLE VIII.—ESTIMATED MINERAL FUEL RESERVES OF THE UNITED STATES AND 1929 PRODUCTION EXPRESSED IN EQUIVALENT TONS OF BITUMINOUS COAL OF 13,000 B.T.U. PER LB. CALORIFIC VALUE.[a]

Description	Billions of net tons		Per Cent Exhaustion	Billions of Net Tons 1929 Production	Recovery, per cent	Years Life at 1929 Rate of Production
	Original Reserve	Reserve, End of 1935				
Anthracite	22	16.5	25	0.074	65[b]	145
Low-volatile bituminous coal[c]	60	......	....	....	70[d]	....
High-volatile bituminous coal[c]	1430	......	....	....	70[d]	....
Subbituminous coal	600	......	....	....	70	....
Lignite	484	......	....	....	70	....
Total, all ranks of coal	2596	2572	1.2	0.613	69	2890
Petroleum	7.2 ?	3.0 ?	58 ?	0.232	100	13
Oil in oil shale	21.2	21.0	0	0	0	0
Natural gas	3.1 ?	1.55 ?	50 ?	0.096	100	16
Total, all fuel	2627	2598	1.4	0.941	...	2130

[a] Heating values used in conversion: Anthracite, 12,700 B.t.u. per lb.
Low-volatile bituminous coal, 14,000 B.t.u. per lb.
High-volatile bituminous coal, 13,500 B.t.u. per lb.
Subbituminous coal, 9500 B.t.u. per lb.
Lignite, 6700 B.t.u. per lb.
Crude petroleum, 6,000,000 B.t.u. per bbl.
Natural gas, 1000 B.t.u. per cu. ft.

[b] In 1921 mining losses were 34.6 per cent, and preparation losses were 4.3 per cent, Report of U. S. Coal Commission, Part II, p. 655 (1925).

[c] Includes some medium volatile coal.

[d] In 1921 a survey of 80 per centof production showed unavoidable losses of 15.3 per cent and avoidable losses of 19.4 per cent making a total recovery of 65.3 per cent, Report of U. S. Coal Commission, Part III, p. 1857 (1925).

States, far from present centers of population. Moreover the bulk of the reserve consists of relatively thin or inaccessible beds which are more difficult to mine. Exhaustion of only 25 per cent of the anthracite deposits has increased considerably the cost of mining. The same situation will develop in the relatively near future with respect to the best bituminous beds, which permit low cost mining.

Advances in fuel technology have contributed greatly to the conservation of coal, but economic conditions in mining have worked against completeness of recovery. The avoidable losses of bituminous coal in time of business activity have been placed at 150,000,000 tons per year. Coal left behind is lost forever, and the prevention of the present wastes is a matter of national concern. Here, also, conservative methods of mining will bring efficient recovery in line with efficient utilization.

FUELS OF TOMORROW

Gas, oil and coal are available today in abundance. There is coal enough for hundreds, and possibly thousands of years; but natural gas and oil obtainable by present methods may be exhausted in less than 100 years, and a shortage of our domestic supply may begin within 10 or 20 years. How will we meet this situation, and what will be the fuels of tomorrow?

An appraisal of present trends and future probabilities indicates that:

1. Coal will continue to be the principal fuel used for the generation of public-utility and major industrial power. Technologic improvements and new hydroelectric power will tend to reduce the consumption of coal; on the other hand, an increasing demand for energy and a decreasing supply of cheap residual oil will increase the amount of coal consumed for power purposes. No material change is expected in either direction in the near future, but in 10 or 15 years the trend will favor increased consumption of coal. Tomorrow's power and central heating plant will burn any kind of coal completely and efficiently. There will be no smoke, no dust, and no sulfurous gases emitted to the atmosphere.

2. No substitute has appeared for metallurgical coke. The coke-oven industry will expand and consume more coal in accordance with metallurgical needs, which are greatly affected by the supply of iron and steel scrap, but relatively few new installations for gas production can be expected in the near future on account of the availability of natural gas. Regulations prohibiting the waste of natural gas and the urge for additional markets will lead to the construction of more long-distance pipe lines from the producing fields to centers of consumption. This gas will find industrial and domestic use, and it will displace oil as well as coal. As natural gas approaches exhaustion, gas from coal will take its place.

3. The convenience and uniformity of automatic heating of homes with gas or oil will continue to attract more users, even at higher costs than

those prevailing today. The insulation of houses has been improved greatly, and future homes will permit higher unit cost of fuel without increasing the total heating bill.

Stoker-fired domestic furnaces, now in their beginning, eventually will give automatic service at a lower cost than that for oil or gas. High- and low-temperature cokes will supplement anthracite as solid smokeless fuel. Smokeless fuels and automatic furnaces will clear the atmosphere of smoke in the better residential districts.

Just how the large low-income group which now burns raw coal will become users of the higher-priced smokeless fuels is not clear. Possibly, the municipality will subsidize plants for the production of smokeless fuels that can be sold for hand-fired furnaces and stoves at a moderate advance above the cost of raw bituminous coal.

On the whole, coal, because it is the cheapest fuel, will continue to contribute the major portion of the fuel used for house-heating and miscellaneous manufacturing, although further displacement by oil and natural gas probably will follow in the next few years.

4. In 1929, 88 per cent of the railroad fuel was coal; since then Diesel locomotives have been adopted by several railroads for light-weight, high-speed passenger trains; an increase in Diesels for such service is expected, but no general change in freight haulage from steam to Diesel power is likely to take place. Further improvements in the over-all efficiency of the steam locomotive and a gradual increase of electrification will retain the use of coal for freight traffic throughout the age of oil and natural gas.

5. Marine transportation is energized by oil. Approximately three-fourths of the marine fuel used in 1936 was oil, and 40 per cent of this oil was used in Diesel engines. This trend will continue. The convenience and economy of Diesel-engine drive for ships and boats is such that its use will continue even after declining production of petroleum requires the production of Diesel fuel from shale or coal.

6. Finally, I come to the most interesting question of all: our motor-fuel supply. Snider and Brooks have predicted the probability of a considerable shortage of domestic petroleum by 1945;[1] and they state that, "The prospect is apparently for a continuation or only a slight modification of the present situation until a shortage of domestic petroleum materializes. Domestic petroleum will then gradually be replaced by imports and substitutes in relative proportions and at price schedules not now predictable beyond a practical certainty that the prices will be appreciably higher than the present ones."

From the very beginning of the automobile industry, recurring threats of shortage of gasoline were met—in the field, by finding new pools and improving production technique, and in the refinery, by increasing yields and making a more efficient product. The end has not been reached. We are just beginning to use scientific methods in extracting oil from the sands, and polymerization and hydrogenation eventually will furnish the means for complete conversion of volatile liquids and heavy petroleum to gasoline.

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[1] L. C. Snider and B. T. Brooks, "Probable Petroleum Shortage in the United States and Methods for Its Alleviation," *Bulletin*, Am. Assn. Petroleum Geologists, Vol. 20, p. 49 (1936).

Although such complete conversion is possible, recent development of the Diesel engine into a light mobile motor is likely to change this trend. The sales of Diesel engines in terms of rated horse-power increased from 750,000 in 1934 to 1,830,000 in 1936. Sixty-four per cent of the horse-power sold in 1936 was applied to portable or automotive equipment and consisted of units of less than 100 hp. Due to the higher thermal efficiency of the Diesel engine and to the fact that it can use the heavier fractions now employed as cracking stock for gasoline, approximately three times the mileage should be obtained from a gallon of crude oil utilized for Diesel fuel than is obtained by conversion to gasoline.

Continuation of present trends toward Diesel power for other purposes also will increase the demand for the heavier oils, resulting in a material shift in future refinery operations. Obviously, economic considerations favor the development of that combination of motor and fuel which converts the greatest portion of the energy in the original petroleum into useful work from the finished fuel.

These improvements, and others yet to come, will add their bit to extending our petroleum reserves. As demand exceeds supply, and prices rise, supplemental fuels of similar characteristics from other sources will come in. These sources are coal, oil shale and vegetable materials.

Increasing demand for Diesel oil and cracking stock will foster coal carbonization, with maximum recovery of liquid by-products. Then as needs increase, hydrogenation of low-temperature tar, shale oil, and coal itself and the synthesis of hydrocarbons from water gas and alcohol from fermentation processes will take over as much of the load as may be required.

It is possible also that producer gas from active forms of solid fuel, such as low-temperature coke and lignite char, will find a place on heavy vehicles operating on steady service, such as trucks and busses. Gas-producer-operated vehicles are being tried in Europe at present; while lacking in convenience and flexibility, the fuel cost is very low.

Reliable information on the cost of making gasoline from coal in British and German plants is not available, but it is believed that it is three or four times the present cost of producing gasoline from petroleum in the United States. These costs will be reduced by further research, but no other liquid motor fuel, whether it be from coal, oil shale, or vegetable matter, can hope to be as cheap as our present petroleum fuels.

Ingenious as they are, the Bergius and Fischer processes are thermally wasteful methods for obtaining power from coal. About 4 tons of coal are required for the manufacture of 1 ton of gasoline by the Bergius method. The gasoline obtained contains only 40 to 45 per cent of the energy in the coal used; and the recovery by the Fischer process is even less. Therefore, from a conservation point of view, it is desirable to delay, as far as possible, the day of making gasoline from coal, not by arbitrary reservation of petroleum for automotive fuel, but by intensive research on promoting the use of coal.

Robinson[1] estimates that 10 to 12 million dollars is spent annually on research and development in the refining of petroleum. Even more should be expended on the preparation and direct utilization of coal. Marvelous strides have been made in power generation in central stations. Little has been accomplished in commercial utilization and domestic heating. Low cost is an important advantage of coal. Research should find a way to obtain heat automatically from coal as effectively as from gas or petroleum. Even today, Diesel engines burning coal dust give some promise of eventual use. Problems due to the ash-forming materials in coal are difficult but not impossible to solve. Such engines, while not suitable for automobiles, would be highly efficient in the generation of power in small stationary plants.

Fortunately, the coal industry is beginning to appreciate the value of research, and a comprehensive program may be expected. It should result in a more rational utilization of the national fuel reserves. Our gas, oil, and coal have energized an industrial civilization on a magnificent scale. Scientists, engineers, and an enterprising business organization have modified these materials to meet our every need; and finally, we are awakening to the need of conserving these resources wisely so that the generations of tomorrow will have better fuels than we of today. Future scientists may unlock undreamed of powers, but we as engineers should plan on the basis of the resources known today.

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ANNUAL REPORT OF THE EXECUTIVE COMMITTEE

The Executive Committee offers the following report on general Society activities for the year ending with this annual meeting:

Membership:

The membership of the Society on June 1, 1937, was 3992. Statistics showing gains and losses in the various classes of membership are as follows:

Class of Membership	Membership		Losses				Additions		Total		
	June 1, 1936	June 1, 1937	Resignations	Dropped	Death	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	1	1	...	..	..	..	..	...	...	...	...
Life or Perpetuity	10	10	...	..	..	..	..	...	...	...	...
Sustaining	10	11	...	..	..	..	1	...	...	1	1
Company, Firm, etc	911	982	15	2	..	6	11	83	23	94	71
Individual, etc	2766	2933	107	45	26	11	14	342	189	356	167
Junior	43	55	4	4	..	9	..	29	17	29	12
Total	3741	3992	126	51	26	26	26	454	229	480	251
Student	204	282	20	89	..	11	..	198	120	198	78

The net gain for the year of 251, compared with the corresponding figures of 129 a year ago and 43 for two years ago, is indicative of the accelerated pace at which recovery in membership has taken place since the losses during the depression. Elections to membership have continued to increase, the figure for this year being 454 compared to 334 and 319 for the past two years. Losses from resignations and delinquencies were the lowest for many years, totaling 177, compared with 183 the previous year and 250 reported two years ago. The number of student memberships increased during the year from 204 to 282, a net gain of 78.

The American Chain and Cable Co., Inc. acquired a sustaining membership during the year, bringing the total of such members to eleven.

Every effort has been made by the Executive Committee to keep in touch with all members who resigned or were dropped in recent years due to financial considerations and to have them resume their membership in the Society. Some 200 members have thus been reinstated in the past several years. While efforts in this direction will be continued, it is clear that we must look to individuals and companies becoming newly interested in the testing of materials to fill up the ranks of membership depleted by losses during the four years of depression.

It is interesting to observe in this connection that many new members are coming into the Society because of expansion of our work into new fields discussed farther on in the report. These new fields should be a fertile source of new members in the next few years.

Again the Executive Committee wishes to express its sincere appreciation of the interest that so many members of the Society have taken in this important work of building up the Society membership. The district committees have been particularly helpful.

The Society has lost the following members through death:

	Date of Membership		Date of Membership
Brazer, G. Herbert	1906	Kern, Walter F.	1925
Brunner, John	1903	Langenberg, Frederick C.	1920
Campbell, William	1903	Neely, P. J.	1936
Chancellor, William C.	1934	Paisley, John W.	1914
Chapin, Joseph E.	1918	Pieraccini, Frank	1933
Doering, R. W.	1934	Ramage, J. C.	1904
Gill, Augustus H.	1905	Roberts, Charles C.	1923
Green, Paul Evans	1919	Sargent, George W.	1910
Gregory, John H.	1906	Schall, Frederick E.	1908
Hall, Charles Ward	1911	Terrell, H. T.	1936
Hammond, George T.	1910	Tung, Teng-Shan	1928
Haswell, John C.	1918	Wadleigh, Francis R.	1908
Hill, Nicholas S., Jr.	1909	Weld, Dana Porter	1934
Ingalls, F. P.	1906		

Among these members are many who contributed greatly to the work of the Society. John Brunner was active for many years in the work of Committee A-1 on Steel and served twice on the Executive Committee, from 1913 to 1915 and again from 1916 to 1918. William Campbell was the dean among non-ferrous metallurgists and was chairman of Committee B-2 on Non-Ferrous Metals and Alloys from its organization in 1909 to his retirement from active leadership in 1934, at which time he was elected honorary chairman. He was a member of the Executive Committee from 1924 to 1926. J. C. Ramage had been active in many committees of the Society and had served on the Executive Committee from 1932 to 1934. A. H. Gill was active in the Society in its earlier days and was chairman of Committee D-2 on Petroleum Products and Lubricants, formerly known as Committee N, from 1906 to 1913. George T. Hammond had been a member of Committee C-4 on Clay Pipe for many years and its chairman since 1929. F. P. Ingalls was one of the pioneer members of Committee D-1 on Paint, Varnish, Lacquer and Related Products. F. C. Langenberg served on a number of the metals committees and on the Executive Committee from 1927 to 1929. F. R. Wadleigh had long been active in the field of coal and coke.

The Executive Committee has elected three honorary members of the Society, of which announcement will be made at the opening session of the annual meeting.

Committee Activities:

The Society completes at this annual meeting a year of exceptional accomplishment in its committee work, both in research and standardization activities. As usual, the annual reports of two administrative committees, E-9 on Research and E-10 on Standards, supplemented by reviews that will appear in the July and October issues of the *Bulletin*, give a general summary of these activities. This report, therefore, will deal briefly with various administrative matters concerning the standing committees.

It has been decided, upon the suggestion of the Executive Committee with the approval of both committees, to merge Committee C-3 on Brick and Committee C-10 on Hollow Masonry Building Units into a single committee on manufactured masonry units. The fields of jurisdiction of the two committees are at present not well defined; there are several masonry products, such as glazed building units, double brick and other forms sometimes designated as tile and sometimes as brick, which might properly be considered to lie within the field of either committee, thus making it difficult satisfactorily to define the scopes of the two committees. Under one committee the question of jurisdiction over these products disappears. Moreover, essentially the same groups of consumers and general interests are involved in the testing and specifying of the products now considered by the two committees. The idea of merging the two committees has met with approval on the part of substantially all who have been active in the committees' work. A reorganization committee under the chairmanship of J. W. McBurney, comprising members from each committee, has been appointed with authority to bring about the formation of the new committee, which may be accomplished at a joint meeting of Committees C-3 and C-10 to be held during the present annual meeting.

The scope of Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys has been extended to include methods of test and specifications for metallic materials used in the manufacture of radio tubes and incandescent lamps, this having come about through requests from the Radio Manufacturers Association that the Society develop such tests and specifications. Most of the metals involved are of the heat-resistance and electrical-resistance type covered by Committee B-4. This, together with work by Committee D-9 on insulating materials for radio, comprises an important extension of A.S.T.M. standardization work into a rapidly developing field.

Expansion of Society work into new materials fields is taking place quite rapidly. At the 1936 annual meeting Committee D-12 on Soaps and Detergents was organized under the chairmanship of H. P. Trevithick and will present its first report to the Society this year. Committee D-18 on Soils for Engineering Purposes was organized last November under the temporary chairmanship of C. A. Hogentogler; at the committee's second meeting last March H. F. Clemmer was elected chairman. The committee is actively engaged in studying various methods of testing soils. The following statement of scope has been adopted:

It will come within the jurisdiction of Committee D-18 to establish methods of sampling and methods of testing soils for engineering purposes, to consider the use of test results in specifications, to select acceptable nomenclature and definitions, and to promote research activity in the general field of properties and behavior of soils for engineering purposes.

It will be the policy of this committee to avoid, in so far as it is possible, control over methods of design of engineering structures and all those features of general practice in the use of soil as an engineering material which may not compromise methods of sampling and testing. It will, however, be considered within the scope of the committee's work to promote by every desirable means the close cooperation of other organizations and committees whose field of endeavor is closely allied to that of soil testing.

Three new committees will be organized during the present annual meeting: Committee D-6 on Paper and Paper Products, Committee C-14 on Glass and Glass Products and Committee D-20 on Plastics. The first of these, Committee D-6, has been under development for the past year under the temporary chairmanship of Roger C. Griffin. It is expected that its initial work will deal with paper testing methods and nomenclature, interpretation and significance of test methods, and methods of test and specifications for certain types of paper products, such as multi-wall paper bags and paper and fiber-board shipping containers. The subcommittee on paper shipping containers was in fact organized during the Chicago Regional Meeting last March, and will deal with paper and fiber-board for containers, as well as the containers themselves, jurisdiction over the latter having been transferred from Committee D-10 to Committee D-6.

The second new committee, C-14 on Glass and Glass Products, was authorized by the Executive Committee last January following a proposal from the American Ceramic Society that an A.S.T.M. committee in this field be organized to develop American standards pertaining to glass and glass products and to act as the channel of American participation in international standardization activities pertaining to glass. Glass is rapidly assuming an important place as a material of engineering and there is a real need for recognized standards. Excellent progress has been made in bringing together a representative personnel of producers, consumers and general interests under the temporary chairmanship of George W. Morey and in planning the immediate organization of six subcommittees that will deal with nomenclature; chemical analysis; chemical, physical and mechanical, and thermal properties; and glass construction block and tile.

The third new committee, D-20 on Plastics, was likewise authorized at the January meeting of the Executive Committee. The desirability of A.S.T.M. work in this rapidly developing field has of course been appreciated for some time, and in recent months a suitable opportunity was presented for organizing a committee to deal particularly with methods of testing plastics. W. E. Emley has been designated as temporary chairman. Much interest has been expressed on the part of both producers and consumers.

It is interesting to note that each of these three materials—paper, glass and plastics—has been studied for some years by Committee D-9 on Electrical Insulating Materials from the viewpoint especially of electrical properties, but including also certain physical and mechanical properties important in the use of these materials for electrical insulation. In each case Committee D-9 will retain jurisdiction over standards involving electrical properties and will cooperate with the new committees in the development of standards for other properties, which will be under their respective jurisdiction.

The Executive Committee has also authorized the organization of a standing committee on thermal insulating materials—another field in which inquiry among members of the Society has indicated the need for standard methods of test. It is intended to exclude the subject of high-temperature thermal insulating materials from the scope of the new committee, since Committee C-8 on Refractories covers that field. Progress towards formal organization is being made.

The Joint Committee on Effect of Phosphorus and Sulfur in Steel, having completed all it could do so far as present commercial demands are concerned, was discharged with the approval and thanks of the various cooperating bodies.

Committee D-1, whose title was changed a year ago from "Preservative Coatings for Structural Materials" to "Paint, Varnish, Lacquer, and Related Products," has developed in connection with certain subcommittee reorganization reported last year[1] a method of dealing with inactive subcommittees in charge of standards that is worthy of special note. It occasionally happens that a subcommittee that has written one or more standards reaches a point where there is no useful work ahead of it. Standing committees in the past have hesitated between two alternatives: to discharge the subcommittee, leaving the standards without an immediate sponsor in the committee; or to continue the subcommittee with nothing to do, which has a somewhat demoralizing effect upon the subcommittee members. Committee D-1 to meet this situation proposes, on discharge of any subcommittee responsible for the development of existing standards, immediately to appoint one member having an active interest in the affairs of Committee D-1 as an "adviser," with the following duties:

(*a*) To review annually all existing standards for which the subcommittee in question, during its life, was responsible.

(*b*) To report at the spring and annual meetings in the same manner as do subcommittee chairmen, upon the standards under their jurisdiction, and to bring to the attention of Committee D-1 any developments that might necessitate alteration of an existing standard for which a discharged subcommittee had been responsible, and to request in such an event the reappointment of a subcommittee to give the matter consideration.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 383—384 (1936).

(*c*) To retain a list of members of the subcommittee at the time of its discharge, in order to communicate with them in the event change in a standard in which they had an interest was to be given consideration.

(*d*) To give consideration to questions pertaining to the standards under their jurisdiction that are received from time to time by the office of the Secretary.

One such adviser has already been appointed in Committee D-1, and it will be interesting to follow the development of this procedure.

Scopes of Committees.—The expansion of the Society's committee work and the unavoidable overlapping of the fields of certain committees, with consequent need of coordination and cooperation between committees, has shown the desirability of adopting formal statements of scope for all committees and of publishing such statements in suitably concise form in the Year Book. During the year scope statements were prepared by the committees which after editing were distributed among all committee officers for final review before publication. The following general statement on the functions of standing committees of the Society will also be published on a separate page in the Year Book preceding the listing of the personnel of standing committees:

> The standing committees of the Society have the broad functions of promoting the knowledge of materials of engineering and the formulation of standard methods of test, specifications, definitions and recommended practices relating to such materials. The advancement of knowledge of the properties of materials is effected through investigations carried on by the committees or sponsored by them. The obtaining of accurate technical information on the characteristics of engineering materials is fundamental and serves as a basis for the standardization work, which comprises the following:
>
> 1. The development of suitable analytical procedures, sampling techniques, and methods of test for determining the constitution, structure or properties of materials.
> 2. The formulation of specifications defining the quality and characteristics of materials.
> 3. The formulation of standard definitions and systems of nomenclature.
> 4. The preparation of recommended practices governing certain methods and processes not ordinarily subject to contract.
>
> The scope of activities of each of the individual standing committees is indicated, particularly as to the materials falling within the jurisdiction of each. In general, the committees are interested in both standardization and investigative work, except in those instances where some limitation is definitely indicated.

Standard Samples.—As reported to the Society a year ago,[1] the Executive Committee has been considering the interest of the Society in the preparation and maintenance of standard samples of materials for use in checking methods of analysis and test—a work that the National Bureau of Standards has been doing for many years. There are a number of materials for which the Society has written standard methods of analysis that are not represented in the Bureau's list of standard samples. In many of these cases standard samples are not considered necessary; in others, technical difficulties or lack of required funds have stood in the way of establishing a standard sample.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36 Part I, p. 46 (1936).

During the year, as a step in the study of this problem, the standing committees were asked, first, whether the standard samples that have so far been prepared by the Bureau are adequate and satisfactory with respect to the materials in which the committees are interested; second, whether there are any materials of interest to the committees for which standard samples have not been prepared by the Bureau; and third, if so, whether the committees will be prepared to lend their support and that of their members in aiding the Bureau to establish such standards. The returns from this questionnaire showed that about twenty committees are interested in standard samples. There was very general agreement that the present samples are satisfactory, and a number of suggestions for new standard samples were made with offers of cooperation from certain of the committees. A report prepared by the Secretary-Treasurer summarizing the results of the questionnaire with recommendations for action was reviewed by a special committee of the Executive Committee headed by W. R. Webster, and the following report of that committee has been adopted by the Executive Committee as an expression of policy to guide the Society in further development of this subject:

> There is evidence that the existing standard samples such as established by the National Bureau of Standards serve a useful purpose, and that there is a substantial demand for additional samples. However, it is not a proper function of the Society to be directly instrumental in extending the list. The Society will in general look to the Bureau of Standards for the development of such standard samples and is prepared to cooperate with the Bureau in this activity.
>
> The demand for such standard samples should come from the individual committees concerned. Preliminary negotiations looking into the feasibility of the standard should be between the standing committees and the Bureau, confirmed by the Secretary-Treasurer after agreement has been reached with the Bureau.
>
> It will be necessary for each committee to provide the material for each sample.
>
> It is desirable wherever standard samples are available that mention be made of this fact in connection with any A.S.T.M. methods of analysis or test where they are applicable. This type of reference could also be supplemented with complete lists of standard samples available, wherever the publication of such list seems to be appropriate.

The Bureau of Standards has given its cordial assurance that it will welcome the help of the Society in the establishment of standard samples, and the matter will be taken up with the Bureau and the interested standing committees. Indeed, one project is now under way, namely, the establishment of a reference sample of silica brick for specific gravity determinations, in which Committee C-8 on Refractories and Committee E-1 on Methods of Testing are cooperating with the Bureau.

Patents Affecting Standards:

The note that has heretofore appeared on certain standards covering materials or processes known or believed to be patented, in accordance with rulings noted in Section 16 of the Regulations Governing Standing Committees, has been modified slightly as to wording, as follows:

> By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

In connection with the revision of this note, some consideration was given to suggestions that it should be made more specific in certain cases where not only composition but such things as processes of casting, fabricating and heat treatment are the subject of patents. It was finally concluded that such elaboration of this note might lead to serious difficulties and that it was better for the note to be general and not too specific.

It has been decided to include the following note on the flyleaf of the Books of Standards and Tentative Standards, Part I of the Proceedings (containing new and revised tentative standards) and the various compilations of standards, in order that all users of Society publications containing standards may be put on notice respecting the Society's policy in these matters:

> Nothing contained in any publication of the American Society for Testing Materials is to be construed as granting any right, by implication or otherwise, for manufacture, sale or use in connection with any method, apparatus or product covered by Letters Patent, nor as insuring anyone against liability for infringement of Letters Patent.

Last year Committee D-2 on Petroleum Products and Lubricants submitted with its annual report, for publication as information, proposed methods of test for kinematic viscosity involving two alternative instruments, one of which was of the suspended level type covered by United States Letters Patent 2,048,305 granted to Dr. Leo Ubbelohde. Under a previous ruling of the Executive Committee,[1] permission to publish these methods was given. After a study of the methods during the year in cooperation with Committee D-2, the Executive Committee has approved with certain stipulations a form of license agreement proposed by the Fish-Schurman Corp., American representative of Doctor Ubbelohde authorized to grant licenses under the patent, under which that Corporation will license the manufacture and sale of the suspended level viscosimeter. The agreement stipulates that viscosimeters manufactured under such license must meet current standards of the Society therefor. The Executive Committee has accordingly approved the submission by Committee D-2 in its current annual report of proposed methods of test for kinematic viscosity including the suspended level viscosimeter, for acceptance by the Society as tentative.

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[1] "Where the patented instrument, appliance or machine concerned is one of two or more alternative instruments, at least one of which is not patented, the Executive Committee will permit reference to such patented instrument in the belief that the interests of the user of the standard are fully protected against an undesirable patent monopoly in that he can use at least one unpatented instrument in accordance with the provisions of the standard."—Sec. 16, Regulations Governing Standing Committees.

Specifications for High-Early-Strength Portland Cement:

The Executive Committee, as directed by action of the annual meeting of the Society last year, reviewed, after approval by Society letter ballot, the provisions of the proposed revised specifications for high-early-strength portland cement pertaining to the proposal for testing such cement containing additions, and with a minor editorial change approved the revised specifications for publication as standard. This action was reported to the Society in the October, 1936, *Bulletin*. Under the terms of this specification[1] as adopted by the Society, Committee C-1 on Cement is empowered to prescribe and carry out tests to determine whether a proposed addition to high-early-strength portland cement "not to exceed 1 per cent of other materials" is "shown not to be harmful." Committee C-1 designated the Cement Reference Laboratory as the agency to make such tests. A form of application for test has been adopted by the Executive Committee after consultation with the Subcommittee on Tests of Additions to Cement of Committee C-1, which embodies terms and conditions of tests that protect the interests both of the Society and the applicant. The current annual report of Committee C-1 contains further reference to this subject.

Proposed Revisions of the By-laws:

The Executive Committee recommends that the By-laws of the Society be revised as indicated below:

Article I, Section 3.—Change the second paragraph to read as follows by the omission of the words in brackets:

A Junior Member shall have the same rights and privileges as a Member, except that he shall not be eligible for office, and his status shall be changed from that of Junior Member to Member at the beginning of the fiscal year next succeeding the date on which he attains the age of twenty-seven years [provided that a Junior Member holding membership at the date of adoption of this Section[1] shall be continued under that status until the beginning of the fiscal year next succeeding the date on which he attains the age of thirty years].

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[1] See Standard Specifications for High-Early-Strength Portland Cement (A.S.T.M. Designation: C 74 – 36), 1936 Book of A.S.T.M. Standards, Part II, p. 4.

[[1] This Section was adopted on September 1, 1919.]

Article VII, Section 2.—Change to read as follows by the omission of the words in brackets:

2. The entrance fees, payable on admission to the Society, shall be $10 for Sustaining Members and Members and $5 for Junior Members. The transfer fee, payable upon transfer from one grade of membership to another, shall be the difference between the corresponding entrance fees [provided that the payment of such transfer fee shall not be required of any Junior Member holding membership at the date of adoption of this section[1]].

[[1] This Section was adopted on September 1, 1926.]

The provisions it is proposed to delete from these two articles are no longer operative since there are in the Society now no Junior Members to whom these provisions apply.

Article III.—Change the last sentence of Section 4 to read as follows by the addition of the italicized words and the omission of those in brackets:

The nominations shall be announced to the members through a notice issued [during the last week in April] *on or before May 5.*

Change Section 5 to read as follows by the addition of the italicized figure and the omission of the figure in brackets:

5. Further nominations, signed by at least 25 members, may be submitted to the Secretary-Treasurer in writing by May [20] *25* and a nomination so made, if accepted by the member nominated, shall be placed on the official ballot.

Change the first sentence of Section 6 to read as follows by the addition of the italicized figure and the omission of the figure in brackets:

An official ballot shall be issued to the members between May [20] *25* and June 1, which ballot shall contain names of nominees made according to the provisions of this Article.

In explanation of these revisions, it is desired to publish early in May the *Bulletin* appearing heretofore at the end of April. Since this *Bulletin* contains the formal announcements of nominations of officers the change in Section 4 is necessary, and this change makes desirable the changes recommended in Sections 5 and 6.

Article VI.—In Section 1 change the third sentence of Paragraph (*c*) to read as follows by the addition of the italicized words and the omission of those in brackets:

During consideration of an action upon the recommendations of a standing committee, [two designated representatives of the standing committee shall serve as members of the Committee on Standards, with full privileges of discussion and vote] *the standing committee may designate representatives to present the recommendations in person, but without right to vote.*

This revision is proposed upon the initiative of Committee E-10 on Standards, as a result of the committee's experience since its organization in 1929. The committee believes that since its function in passing upon a standard proposed by a standing committee is primarily the judicial one of determining whether all requirements relating to the preparation and submission of the standard have been met and whether a substantial consensus of the standing committee has been reached, rather than to pass upon the technical merits of the standard, it is incompatible with this function that the two designated representatives of the standing committee provided for at present should have the privilege of voting. It is at the same time desirable that representatives of the standing committee present the recommendations in person. While the Executive Committee concurred in this position, it first submitted the proposal to the officers of all standing committees for comment, including the membership of Committee E-5 on Standing Committees. All replies received from committee officers were favorable, and the Executive Committee accordingly asks the Society to make this change in the standardization procedure. If approved, an identical change in Section 15 (*c*) of the Regulations Governing Standing Committees will be made.

To conclude, the Executive Committee recommends that the above-cited proposed revisions of the By-laws be referred to letter ballot of the Society for adoption.

District Committees:

The eight district committees of the Society have been functioning for two years under the revised charter promulgated by the Executive Committee in 1935. Experience indicates that under this charter the district committees can do much useful work in the interest of A.S.T.M. Most of the district committee activities have been reported currently in the *Bulletin*, including an account of local and district meetings held under their sponsorships, to which further reference is made in the following section of this report. Certain other activities do not lend themselves to description in this way but are nevertheless effective and valuable, including membership work in which several of the committees have been actively interested.

During the Regional Meeting of the Society in Chicago in March, 1937, a conference of officers of district committees was arranged, at which a number of items of mutual interest to the district committees were discussed and suggestions made for consideration during the coming year. It was felt that this conference was quite successful and it is planned to make it a feature of succeeding Regional Meetings.

In the summer of 1936 W. M. Barr, formerly a member of the Executive Committee, visited the Southern California and Northern California District Committees, reviewed their activities and discussed with the committees a number of problems that had arisen in their work. This personal conference was extremely helpful to both the Executive Committee and these two far-removed district committees.

Meetings and Local Activities:

Since the thirty-ninth annual meeting last June at Atlantic City, the eighth Regional Meeting was held at the Palmer House, Chicago, March 2 and 3, 1937, under the auspices of the Chicago District Committee. The technical features of the meeting were a Symposium on Corrosion Testing Procedure, developed under the auspices of Committees A-5 on Corrosion of Iron and Steel and B-3 on Corrosion of Non-Ferrous Metals and Alloys, and a Symposium on Lubricants, developed under the auspices of Technical Committee B on Motor Oils of Committee D-2 on Petroleum Products and Lubricants. The first symposium comprised one session on March 2; the second comprised two sessions on March 3. The attendance of about 350 at each session was indicative of the keen interest in the subjects discussed. The Lubricants Symposium papers with discussion are now being edited for publication as a separate Regional Meeting volume. Discussion on the corrosion testing papers is being continued at one of the sessions of the present annual meeting, after which these papers and discussions will be taken in hand for publication. The March, 1937, *Bulletin* gives an account of the various activities associated with this very successful Regional Meeting.

The Spring Group Meetings of Committees were also held at Chicago, March 1 to 4, inclusive. About 150 meetings of main committees, subcommittees and sections were held; the total registered attendance, exclusive of visitors to the two symposiums, was 588. The committee meetings were uniformly well attended and the committees accomplished a great deal of work.

A number of interesting local meetings have been held this year under the auspices of the district committees. Accounts of these meetings have appeared currently in the *Bulletin* but it is desired to record the meetings here with an expression of appreciation from the Executive Committee for this helpful form of activity. On September 17, 1936, members of the Society in the Northern California District participated in a combined dinner meeting in San Francisco with the Purchasing Agents Association of Northern California, at which Welding was the topic of discussion. On January 28, 1937, a meeting was held in Cleveland under the joint auspices of the Cleveland District Committee and the Cleveland Section of the American Society of Mechanical Engineers, at which several papers on The Making of Specifications were presented, including one by President A. C. Fieldner. The four papers were published either in full or in abstract in the March *Bulletin*. On February 25, 1937, a dinner meeting was held in Pittsburgh under the auspices of the Pittsburgh District Committee, at which the subject of Glass was featured. The three papers read at the meeting were likewise published in the March *Bulletin.*

On April 5, 1937, the Philadelphia District Committee sponsored a Symposium on Wear of Metals, at which six papers were presented at afternoon and evening sessions held at the Engineers' Club of Philadelphia. A dinner was held between the sessions. These papers developed a great deal of worth-while information and together with the discussion will be published as a separate technical pamphlet. On April 8, 1937, a meeting was held in Detroit under the auspices of the Detroit District Committee, at which the principal address was on Recent Progress in Rubber Technology, which was published in the April *Bulletin.*

The President attended all of these local meetings, save that held in San Francisco, and brought appropriate greetings from the Society to the members and guests present. These meetings have served excellently the dual purpose of affording an occasional opportunity for members of the Society in a given locality to get together as A.S.T.M. men for the discussion of some materials topic, and of acquainting other engineers and scientists who attend as guests with the purposes and activities of the Society. They are proving to be quite an effective means of promoting the welfare of the Society, and the district committees are to be commended for their initiative in holding them and in putting so much of technical value into the programs.

Finally, the New York District Committee, suitably enlarged for the year into a New York Committee on Arrangements, is acting as host to the Society at the present annual meeting in New York City. Through the organization of committees on Entertainment, Program, Finances, Publicity, Exhibit, Membership and Ladies Entertainment, these New York members have been working for months to make this annual meeting one that the Society will long remember. No efforts are being spared to make the meeting a pleasant one for both the members and their ladies, as is evident from the entertainment programs that have already been announced. The thanks of the Executive Committee are tendered to each member of the New York Committee.

Publications:

The publication work of the Society is in the very nature of things its most important single activity, since the publications represent the result of all the efforts of our committees and the members who present papers and discussions. It has become more or less trite to refer to this work as active; it is no exaggeration to say, however, that the publications of the Society are growing both in volume and in industrial and technical value with each year. The year that closes with this annual meeting has surpassed any previous year in the volume of publications issued.

The following regular publications were issued and distributed to the members:

1936 Proceedings: Part I (Committee Reports), 1259 pages; Part II (Technical Papers), 852 pages; total 2111 pages, 4250 copies each part; in addition to preprints of reports and papers totaling approximately 1150 pages.

1936 Book of A.S.T.M. Standards; Part I (Metals), 916 pages, two insert plates; Part II (Non-Metallic Materials), 1503 pages, four insert plates; total 2419 pages, 7200 copies each part.

A.S.T.M. Methods of Chemical Analyses of Metals, 260 pages, 1500 copies (a new publication).

1937 Index to A.S.T.M. Standards and Tentative Standards, 120 pages, 12,000 copies.

Year Book, 246 pages, 2750 copies.

Six issues of the A.S.T.M. *Bulletin* were published. The Book of A.S.T.M. Tentative Standards (1422 pages, 1800 copies) and the Symposium on High-Strength Constructional Metals (132 pages, 2000 copies), the latter containing the papers and discussions at the Pittsburgh Regional Meeting in March, 1936, were published and furnished to members on order.

A comparative statement of the volume of regular publications for the past four years follows:

	1933, PAGES	1934, PAGES	1935, PAGES	1936, PAGES
Proceedings	1914	2288	2279	2111
Book of A.S.T.M. Standards	2348	....	....	2419
Supplement to Book of A.S.T.M. Standards	....	224	216	....
A.S.T.M. Methods of Chemical Analyses of Metals	....	....	....	260
Index to A.S.T.M. Standards and Tentative Standards	124	....[a]	142	160
Year Book	248	251	256	246
Book of A.S.T.M. Tentative Standards	1164	1288	1628	1422
Regional Meeting Symposium	121	119	156	132
Report on Significance of Tests of Concrete and Concrete Aggregates	....	127	....	....
	5919	4297	4677	6750

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[a] Date of publication of Index changed to January 1 of succeeding year.

In pursuance of recommendations made by Committee E-6 on Papers and Publications and after conferring with a number of the committees and members interested, it was decided to publish in a separate volume all the methods of chemical analysis of metals, both standard and tentative, omitting them from the Books of Standards and Tentative Standards and the current Proceedings, and to make this separate volume available without charge to all members who request a copy. This move seems to have met with instant approval from those members who are especially interested in having these methods readily available in most convenient form. About 650 members have requested copies and in addition approximately 700 copies have been sold to members and others. No criticisms of this method of publication have been received, but final decision on its permanence must be deferred until more experience has been gained with the publication of revised editions in the future.

In addition to the above there was published last March 1200 copies of the 200-page five-year Index to the Proceedings covering volumes from 1931 to 1935, inclusive. An important special technical publication was issued comprising the papers and discussions in the Symposium on Radiography and X-ray Diffraction Methods held during the 1936 annual meeting; 2000 copies of the 358-page book were published in January.

During the past year the Society also published the following compilations of standards:

A.S.T.M. Standards on Petroleum Products and Lubricants (Committee D-2) 378 pages, 3750 copies, published annually.

A.S.T.M. Standards on Coal and Coke (Committee D-5), 147 pages, one insert plate, 1500 copies, published biennially.

A.S.T.M. Standards on Electrical Insulating Materials (Committee D-9), 335 pages, one insert plate, 700 copies, published annually.

A.S.T.M. Standards on Textile Materials (Committee D-13), 301 pages, 1500 copies, published annually.

The three other compilations of standards that the Society has published, which complete this series, are those dealing with Refractory Materials (Committee C-8), Rubber Products (Committee D-11), and Paints (Committee D-1) which were last published in 1935. These having been published heretofore on the biennial basis, it is expected that they will be reprinted in revised form this fall.

Other reprints and special publications printed since the last annual meeting include:

A.S.T.M. Manual on Presentation of Data (Committee E-1), 74 pages, 2000 copies.

Marburg Lecture "Developing American Glass" by Arthur L. Day, 16 pages, 1000 copies.

Data on Nominal Creep Strength of Steels at Elevated Temperatures (Committee A-1), 34 pages, 1000 copies.

Report of Committee A-5 on Corrosion of Iron and Steel, 16 pages, three insert plates, 1000 copies.

This year it was possible to carry on vigorously the expansion of the A.S.T.M. *Bulletin* along lines that have been announced in previous reports of the Executive Committee to the Society. Several new features have been introduced in the *Bulletin*, as have doubtless been observed by the readers. The most important of these is the inclusion of papers of the following types: (1) Papers of a non-technical nature dealing with matters within the field of the Society and of current interest; (2) papers describing exploratory research which has not progressed to the point where publication of a technical report in the *Proceedings* is justified; (3) papers describing a new application of an established test method or a technical description of a new testing instrument; and (4) papers offered for publication in the *Bulletin* by any of the standing committees for the purpose of eliciting discussion more expeditiously and with greater continuity than can be obtained from annual publication. This list is not to be taken as excluding papers in other categories. During the past year a number of papers have been published in the *Bulletin*, including various ones presented at district meetings. For the first time several papers appearing on the program of an annual meeting will be published this year in the *Bulletin*. An increase in the budget appropriations for the *Bulletin* has made this expansion possible, although the increase in advertising income in the past year has gone far towards meeting the additional costs.

Development of the *Bulletin* along these new lines will, of course, be done under the direction of Committee E-6 on Papers and Publications, for which purpose a Special Committee on Bulletin has been appointed consisting of W. E. Emley, chairman, C. L. Hippensteel and J. C. Pearson.

Finances:

Report for the Fiscal Year 1936.—The annual statement of the finances of the Society follows in the report of the auditors for the fiscal year January 1 to December 31, 1936.

Total receipts from dues and entrance fees in 1936 were $74,711.01, a net increase of about $4850 over the preceding year. Income from this source is, of course, increasing with the growth of membership that set in with 1935. Receipts from sales of publications totaled $45,290.29, which is by far the largest in the history of the Society. Each year for the past several years has seen a marked increase in this source of income, which is both fortunate and gratifying; fortunate in that it has offset the loss of income from reduced membership, and gratifying in that it demonstrates most convincingly the intrinsic value of the Society's publications and the growing industrial demand for them. Income from this source is materially above the best records of pre-depression days and last year comprised slightly over one-third of the total income. Miscellaneous receipts amounted to $11,804.52, which includes substantially increased income from advertising in the Society *Bulletin* and the Index to Standards and Tentative Standards. Total operating receipts were $131,805.82. Total operating disbursements with all current bills paid, as detailed in the auditors' report, were $125,012.33, leaving a favorable balance between operating receipts and operating disbursements of $6793.49, which was added to the reserve funds of the Society.

The report of the auditors gives the balance sheet of assets and liabilities, including special and designated funds (that is the Research Fund, Medal and Lecture Fund and Committee Funds); statement of receipts

REPORT OF THE AUDITORS FOR THE FISCAL YEAR, JANUARY 1, 1936, TO DECEMBER 31, 1936

PHILADELPHIA, January 8, 1937

MR. C. L. WARWICK, *Secretary-Treasurer*
AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pa.

Dear Sir:

We have examined the books and accounts of your Society for the year ended December 31, 1936. The books and accounts were found correct and in satisfactory condition.

We have prepared the annexed Balance Sheet as of December 31, 1936, and Statement of Cash Receipts and Disbursements for the twelve months ended that date, together with other supporting schedules. The schedules are in agreement with the records of your Society.

Investments owned were verified by actual inspection and count and found to be intact and in agreement with the schedule of securities owned as detailed. We verified the collection of interest on investments owned for the year 1936, and found it fully accounted for.

Respectfully submitted,

(Signed) JOHN HEINS AND CO.

BALANCE SHEET AS OF DECEMBER 31, 1936

(Including Special and Designated **Funds**)

ASSETS

General Funds:				
Current: Cash	$20 943.82			
Less checks drawn as of December 31, 1936—Cost of *Proceedings* in hands of Secretary-Treasurer that date.	14 000.00			
		$6 943.82		
Investments (Market Value, $35,375.63)—Cost		29 767.23		
Accounts Receivable		14 668.66		
Total Current Assets—General Fund			$51 379.71	
Furniture and Fixtures			6 915.95	
A.S.T.M.-A.S.M.E. Symposium on Effect of Temperature: Special Account for Financing Book			1 420.25	
Total General Funds				$59 715.91
Special and Designated Funds:				
Cash: A.S.T.M. Research Fund		$1 568.49		
Dudley Medal and Marburg Lecture Fund		571.17		
Committee Funds		7 911.22		
			$10 050.88	
Investments:				
A.S.T.M. Research Fund (Market Value, $9,626.87)—Cost		$10 082.91		
Dudley Medal and Marburg Lecture Fund (Market Value, $5,918.75)—Cost		6 625.00		
Committee Funds (Market Value, $6,377.50) Cost		$6 000.00		
			22 707.91	
Total Special and Designated Funds				32 758.79
Total Assets				$92,474.70

LIABILITIES

General Funds:			
Current: Accounts Payable—			
Committee A-1	$82.47		
Committee C-1	31.27		
International Association for Testing Materials	12.85		
Advance Collections	2 498.03		
Total Current Liabilities—General Fund		$2 624.62	
Life Membership Fund	$3 005.20		
Headquarters Fund	1 041.56		
Publication Fund	3 015.00		
Surplus	50 029.53		
		57 091.29	
Total General Funds			$59 715.91
Special and Designated Funds:			
A.S.T.M. Research Fund		$11 651.40	
Dudley Medal and Marburg Lecture Fund		7 196.17	
Committee Funds		13 911.22	
Total Special and Designated Funds			32 758.79
Total Liabilities and Funds			$92 474.70

COMPARISON OF GENERAL FUNDS FOR FISCAL YEARS 1932–1936, INCLUSIVE

At Close of Fiscal Year	Assets				Liabilities			
	Cash	Investments[a]	Accounts Receivable and Miscellaneous	Furniture and Fixtures	Accounts Payable and Miscellaneous	Reserve for Book of Standards	Miscellaneous Funds[a]	Surplus
1932	$5 945.05	$49 035.80	$14 834.01	$6 301.67	$2 077.61	$11 743.20	$19 229.55	$43 066.17
1933	7 869.45	38 266.50	16 739.78	6 092.51	3 178.98	....	19 008.68	46 780.58
1934	3 792.12	38 924.05	9 767.35	8 010.84	2 204.99	5 023.44	7 061.76	46 204.17
1935	10 514.73	36 180.42	9 220.39	7 489.70	2 695.60	10 023.44	7 061.76	43 624.44
1936	6 943.82	29 767.23	16 088.91	6 915.95	2 624.62	....	7 061.76	50 029.53

[a] The funds reported in this column for 1932 and 1933 include the principal of the A.S.T.M. Research Fund, which beginning with 1934 is no longer included under "General Funds."

RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD JANUARY 1, 1936, TO DECEMBER 31, 1936

Cash balance, January 1, 1936			$10 514.73
RECEIPTS			
Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$69 424.77		
Past Dues	949.54		
Advance Dues	1 149.30		
Income, Life Membership Fund	150.00		
Entrance Fees	3 037.40		
Total Dues		$74 711.01	
Sale of Publications:			
Separate Standards	$5 384.61		
Book of Standards	9 584.28		
Book of Standards (Members, both parts)	4 463.99		
Book of Tentative Standards	5 574.25		
Methods of Chemical Analysis of Metals	77.15		
Proceedings	3 253.47		
Selected Standards for Students	666.50		
Special Reprints and Miscellaneous	16 286.04		
Total Sale of Publications		45 290.29	
Miscellaneous:			
Binding (Members)	$494.00		
Advertising, Bulletin	5 108.70		
Advertising, Index	1 372.50		
Interest on Deposits and Investments	2 283.22		
Registration and Other Fees, Annual Meeting	1 266.00		
A.S.T.M. Exhibit, Annual Meeting	450.00		
Committee A-1 for Technical Assistant	752.00		
Miscellaneous	78.10		
Total Miscellaneous Items		11 804.52	
Total Operating Receipts (Budgeted)		$131 805.82	
Non-Operating Receipts (Not Budgeted):			
Investments, Matured or Sold	$14 411.00		
Excess Remittances	126.05		
Accrued Interest	59.62		
International Association for Testing Materials, Dues and Publications	724.00		
Symposium on Effect of Temperature	324.11		
Committee C-1 for Technical Assistant	1 000.00		
Seaview Golf Club (see Disbursements)	20.20		
Miscellaneous	9.40		
Total Non-Operating Receipts		16 674.38	
Total Receipts			148 480.20
Carried forward			$158 994.93

Brought forward			$158 994.93

DISBURSEMENTS

Operating Disbursements (Budgeted):			
Publications:			
Year Book	$1 829.56		
Book of Standards	8 256.19		
Book of Tentative Standards	3 517.86		
Methods of Chemical Analysis of Metals	900.00		
Proceedings	19 161.68[a]		
Preprints	2 432.38		
Regional Meeting Papers	604.46		
Bulletin and Circulars to Members	5 262.22		
Separate Standards	2 956.74		
Index to Standards and Tentative Standards	1 641.22		
Reprints (Reports, Papers, Symposiums, etc.)	7 273.91		
Miscellaneous Printing (Authors' Reprints, etc.)	301.84		
Total Disbursements, Publications		$54 138.06	
Salaries		46 838.20	
General Office Expenses		10 990.51	
Expenses, Standing and District Committees		3 002.86	
Expenses, Meetings and A.S.T.M. Exhibit		2 577.38	
Traveling Expenses, Administrative and Special Committees		1 429.42	
American Standards Assn. and Sectional Committees		1 000.00	
Rent, Headquarters		4 200.00	
Furniture and Fixtures		414.25	
Miscellaneous		421.65	
Total Operating Disbursements (Budgeted)			$125 012.33
Non-Operating Disbursements (Not Budgeted):			
Book of Standards Reserve	$10 023.44		
Investments	12 104.83		
Accrued Interest	59.62		
Refund of Excess Remittances	126.05		
International Assn. for Testing Materials	728.00		
Symposium on Effect of Temperature	23.45		
Technical Assistant, Committee C-1	1 453.19		
Principal A.S.T.M. Research Fund	2 500.00		
Seaview Golf Club (see Receipts)	20.20		
Total Non-Operating Disbursements		$27 038.78	
Total Disbursements			$152 051.11
Cash Balance December 31, 1936			$6 943.82

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[a] This amount includes $14,000.00 in the form of checks drawn against cost of *Proceedings*, but not actually paid on December 31, 1936, pending final completion of the publication work.

In addition to the general funds of the Society, the Secretary-Treasurer has on hand the following funds:

A.S.T.M. RESEARCH FUND

Principal Account:		Total	Invested	Uninvested Cash
Balance January 1, 1936 as Reported		$7 406.00	$7 363.41	$42.59
Receipts—Proceeds Sale of Securities			−1 552.50	+1 552.50
Contributions from Members	$300.00			
Contribution A.S.T.M. Entrance Fees	2 500.00			
Contribution from Joint Committee on Investigation of the Effect of Phosphorus and Sulfur in Steel	373.71	+3 173.71		+3 173.71
		10 579.71	5 810.91	4 768.80
Disbursements—Purchase of Securities			+4 312.50	−4 312.50
			10 123.41	456.30
Loss on Sale of Securities		−40.50	−40.50	
Balance December 31, 1936		$10 539.21	$10 082.91	$456.30

The invested portion of the fund refers to cost of investments held.

Income Account:

Cash Balance, January 1, 1936	$1 316.51		
Receipts			
Interest on Deposits and Investments	567.08		
		$1 883.59	
Disbursements			
Joint Research Project with University of Illinois, on Speed of Testing	$600.00		
Research Committee on Yield Point	11.82		
Joint Committee on Boiler Feed Water Studies	100.00		
Accrued Interest on Securities Purchased	59.58		
		$771.40	
Cash Balance, December 31, 1936			$1 112.19

DUDLEY MEDAL AND MARBURG LECTURE FUND

Balance, January 1, 1936:			
Principal—Investments (At Cost)	$6 625.00		
Income—Cash	619.42		
		$7 244.42	
Receipts			
Interest on Investments		330.00	
			$7 574.42
Disbursements			
Honorarium, 1936 Lecturer		$200.00	
Cost of 1936 Medal		80.00	
Engrossing 1936 Lecture Certificate		3.25	
Miscellaneous		95.00	
			378.25
Balance, December 31, 1936:			
Principal—Investments (At Cost)		$6 625.00	
Income—Cash		571.17	
			$7 196.17

COMMITTEE FUNDS

Committee A-1 on Steel	$363.81	
Committee A-5 on Corrosion of Iron and Steel	1 954.37	
Committee A-5, Subcommittee X on Embrittlement Investigation	131.34	
Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys	3 201.91	
Committee B-6 on Die-Cast Metals and Alloys	198.94	
Committee C-1 on Cement	973.85	
Committee C-1, Cement Reference Laboratory	1 732.09	
Committee C-1 on Cement, Studies of Additions to Cement	2 412.30	
Committee C-9 on Concrete and Concrete Aggregates	650.97	
Committee C-10 on Hollow Masonry Building Units	264.18	
Committee C-18 on Natural Building Stones and Slate	160.01	
Committee D-1 on Preservative Coatings for Structural Materials	35.04	
Committee D-13 on Textile Materials	101.00	
Committee D-14 on Screen Wire Cloth	14.47	
A.S.A. Sectional Committee on Specifications for Cast-Iron Pipe and Special Castings	612.79	
A.S.A. Sectional Committee on Classification of Coals	1 104.15	
Total Committee Funds		$13 911.22
Accounted for as follows:		
Investments (At Cost)	$6 000.00	
Cash Balance, December 31, 1936	7 911.22	
		$13 911.22

INVESTMENTS, DECEMBER 31, 1936

Par Value:	GENERAL FUNDS	*Book Value*	*Market Value*[1]
$9 000	Baltimore & Ohio R.R. General and Refunding Bonds, Series F, 5's, due 1996	$9 106.55	$8 032.50
10 000	New York Central R. R. Refunding and Improvement Bonds, 5's, due 2013	5 108.55	10 112.50
4 000	New York, Chicago & St. Louis R. R. Refunding and Mortgage Bonds, Series C, 4½'s, due 1978	2 805.61	3 760.00
3 000	Southern Pacific, Oregon Lines Bonds, Series A, 4½'s, due 1977	2 576.69	2 985.00
3 000	Wheeling Steel Corp., First Mortgage Sinking Fund Bonds, Series A, 4½'s, due 1966	3 045.00	3 067.50
3 000	Republic Steel Corp., General Mortgage Bonds, Series B, 4½'s, due 1961	3 000.00	2 985.00
2 000	North Station Industrial Bldg., Inc., First Mortgage Sinking Fund Bonds, 5's, due 1962	1 850.00	1 770.00
500	Saguenay Power Co., Ltd., First Mortgage Sinking Fund Bonds, Series A, 4¼'s, due 1966	500.00	519.38
5 000	50 shares Great Northern Ry. Preferred Stock	1 774.83	2 143.75
$39 500		$29 767.23	$35 375.63
	A.S.T.M. RESEARCH FUND		
$1 500	Baltimore & Ohio R. R. General and Refunding Bonds, Series F, 5's, due 1996	$1 511.25	$1 338.75
2 000	New York, Chicago & St. Louis Refunding and Mortgage Bonds, Series C, 4½'s, due 1978	1 400.26	1 880.00
1 000	Southern Pacific, Oregon Lines Bonds, Series A, 4½'s, due 1977	858.90	995.00
2 000	First Mortgage Real Estate Bonds, 1315–1317 Spruce St., Philadelphia, 6's, due 1945	2 000.00	1 200.00
3 000	North Station Industrial Bldg., Inc., First Mortgage Sinking Fund Bonds, 5's, due 1962	2 812.50	2 655.00
1 500	Saguenay Power Co., Ltd., First Mortgage Sinking Fund Bonds, Series A, 4¼'s, due 1966	1 500.00	1 558.12
$11 000		$10 082.91	$9 626.87
$50 500	Carried foreward	$39 850.14	$45 002.50

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[1] Market Values, except on First Mortgage Real Estate 1315–1317 Spruce St., 6's, 1945, on which there was no quotation, and North Station Industrial Bldg., Inc., First Mortgage Sinking Fund, 5's, 1962, on which a quotation was obtained from the brokers, were taken from the *Wall Street Journal* of January 2, 1937.

INVESTMENTS (*Continued*)

Par Value			
$50 500	Brought forward	$39 850.14	$45 002.50
	DUDLEY MEDAL AND MARBURG LECTURE FUND		
$ 500	Baltimore & Ohio R. R. General and Refunding Bonds, 6's, due 1995	$550.00	$503.75
6 000	Baltimore & Ohio R. R. General and Refunding Bonds, 5's, due 1995	6 075.00	5 415.00
$6 500		$6 625.00	$5 918.75
	COMMITTEE FUNDS		
$5 000	U. S. Treasury Bonds, 2¾'s, due 1947	$5 000.00	$5 331.25
1 000	U. S. Treasury Bonds, 2⅞'s, due 1960	1 000.00	1 046.25
$6 000		$6 000.00	$6 337.50
$63 000		$52 465.14	$57 298.75

and disbursements in which the items are classified into "operating" (budgeted) and "non-operating"; details of special funds; investment of Society funds, both book and market values being given; and a comparison of the financial condition of the Society at the close of the past five years. The surplus in the general fund account, exclusive of special and designated funds, is $50,029.53, which compares with the figure for a year ago of $43,624.44.

No account has been taken in the accompanying financial statement of the assets of the Society in the form of publications in stock, with the exception of the cost value, $1420.25, of the copies still in stock of the joint A.S.M.E.-A.S.T.M. Symposium on Effect of Temperature on the Properties of Metals purchased by the Society from a joint account described in a previous report;[1] this amount is included in the statement of assets. The inventory of technical publications on June 1, 1937, may be summarized approximately as follows:

	Number of Copies
Year Book	288
Proceedings	4 150
Index to Proceedings, 5 parts, total	1 986
1936 Book of A.S.T.M. Standards – Part I, Metals	2 142
1936 Book of A.S.T.M. Standards – Part II, Non-Metals	2 253
1936 Book of A.S.T.M. Tentative Standards	0
Methods of Chemical Analyses of Metals	150
Index to Standards and Tentative Standards	1 930
Selected Standards for Students in Engineering	221
Reprints of 513 Standards (approximate)	37 000
Symposium on Effect of Temperature on the Properties of Metals	387
Special Reprints (approximate)	21 500

1937 Finances.—Society operations for 1937 have been based on a budget of receipts which anticipates the growth of membership and the continued demand for the Society publications. Receipts from dues and entrance fees are estimated at $79,300; receipts from sales of publications are estimated at $48,150; miscellaneous receipts, including interest on investments, advertising in the *Bulletin* and Index to Standards, and gross receipts from the Fourth Exhibit of Testing Apparatus and Equipment held in conjunction with the present annual meeting, are estimated at $14,800, bringing the total of estimated current receipts to $142,250. Disbursements have been estimated at $138,700, leaving $3550 for contingencies. The budget of disbursements provides fully for all of the Society's normal operating expenses, including all regular and special publications. Special provision has been made for publishing an enlarged *Bulletin*, as mentioned earlier in the report.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 53 (1932).

The Executive Committee reviews the budget of receipts and disbursements quarterly and makes any adjustments in appropriations for operating expenses that may be needed in the light of actual receipts. Experience of the first five months with respect to income from all sources indicates that the above estimate of income for the year is a properly conservative one.

In April the Executive Committee transferred $2000 from general surplus to the Headquarters Fund, a transaction that will be reflected in the auditors' report for 1937. The growth of the Society's work involves growth of staff and in due time expansion of Society headquarters. It seems desirable to the Executive Committee to begin now to make some provision for possible extraordinary expenses in the future in relation to the maintenance of suitable Society headquarters.

Research Fund:

In the report a year ago[1] the Executive Committee reported that it had transferred the sum of $2500 from general reserve funds to the principal of the A.S.T.M. Research Fund, this representing part of the entrance fees in recent years which had been temporarily held in a contingency reserve fund. This transaction is recorded in the accompanying auditors' report in the section reporting "non-operating disbursements." In January, 1937, a second sum of $2500 was likewise transferred to the principal of the Research Fund and will be recorded in the auditors' report for the current year.

It is with pleasure that the Executive Committee announces to the Society the substantial gift to the principal of the Research Fund made by Past-President Frank O. Clements, in the form of shares of common stock of the General Motors Corp. valued in the Society's books at approximately $6500. (This gift, having been made in February, 1937, does not appear in the present auditors' report.) Under the terms of the deed of gift the income from this sum will be paid to an annuitant during her lifetime, after which the income reverts without restriction to the Research Fund. This very substantial gift brings the principal of the Fund up to approximately $20,000. Expressed in another way, the principal of the Research Fund has practically doubled within the past year.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 60 (1936).

Doctor Clements' gift is in keeping with the spirit of the Special Research Fund Committee comprising himself as chairman, K. G. Mackenzie and H. F. Moore, devoted to building up the Research Fund. The committee is for the time concentrating its efforts on inducing members to make bequests to the Fund in their wills. It is to be hoped that this gift will inspire others to augment the Research Fund as their interest in the Society may dictate and their means will permit. An active Research Fund of reasonable proportions will greatly multiply the usefulness of the Society.

Cooperative Activities:

The Society's contacts with the American Standards Association constitute one of its most important cooperative activities. During the past year the principal work has been more or less routine in nature, involving the submission of new and revised standards to the A.S.A. for approval as American Standard or American Tentative Standard. The annual report of Committee E-10 on Standards presents this work in further detail.

J. A. Capp has been re-appointed as one of the Society's representatives on the Standards Council of A.S.A. for the term of three years ending December 31, 1939. The Assistant Secretary of the Society has been re-appointed as alternate for a term of one year.

The Executive Committee has authorized a study of the relationship of the Society to laboratory service performed by a wide range and variety of laboratories using the test methods and specifications of A.S.T.M., which is being undertaken on the recommendation of an active member of the Society who has long been identified with commercial laboratory work and service. The following committee has been named to conduct this study:

T. G. Delbridge, Vice-President of the Society, Atlantic Refining Co. (chairman)
H. H. Craver, Pittsburgh Testing Laboratory
E. C. Crittenden, National Bureau of Standards
D. E. Douty, United States Testing Co.
F. M. Farmer, Electrical Testing Laboratories
H. H. Morgan, Robert W. Hunt Co.
F. E. Richart, University of Illinois

International Relations:

During the past year the Society has maintained contact with the activities of the International Association for Testing Materials, principally through W. H. Fulweiler, American Member of the Permanent Committee and Vice-President of I.A.T.M. The Second International Congress of the I.A.T.M. was held in London April 19 to 24, 1937, under the auspices of the British Branch, I.A.T.M., at which some 200 papers were presented, about twenty of them being by American authors secured in part directly by the Congress officials and in part through the offices of Mr. Fulweiler. Announcements of the Congress were mailed to Society members and were published in the Society *Bulletin*. W. E. Emley and R. L. Templin were designated as official representatives of the Society to the Congress; their report will be considered by the Executive Committee at its next meeting. It is understood that a reorganization of the International Association was effected at the Congress, eliminating individual memberships and the paid secretariat, and limiting the essential activities of the Association to the holding of periodic congresses sponsored by national testing societies and financed primarily by them. The details of reorganization will in due time be officially transmitted to the Society for consideration.

The Executive Committee has given careful consideration to the position the Society should take in the matter of the financial deficit of the International Association, which was incurred in part through the publication of four volumes of "First Communications" comprising preliminary reports and papers for the 1931 Zurich Congress[1] and in part through the difficulties experienced in selling the Proceedings of the Zurich Congress during the years of world-wide depression that followed that Congress. Subscriptions solicited from American members of the I.A.T.M. were applied to the deficit but fell far short of the proportion of the deficit that had been "apportioned" to this country. In consideration of substantial contributions that have been made by the national materials testing societies or groups in many other countries supporting the I.A.T.M., and in further consideration of assurance that the London Congress would be placed upon a satisfactory financial basis and the International Association would be reorganized along the lines just mentioned, the Executive Committee has recently contributed from the reserve funds of the Society the sum of $1000 towards the deficit, with the understanding that this is the full extent to which we are prepared to contribute. The cause of international discussions of the properties and tests of materials is a worthy one, deserving of American interest and reasonable financial support, and it is this spirit that has actuated the Executive Committee in this matter.

Other international activities of the Society are briefly noted. International standardization is considered through committees of the International Standards Association (ISA) and the Society has named various American members of ISA committees for which the American Standards Association acts as the American clearing house. The current report of Committee E-10 on Standards will refer to any significant developments in this field.

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[1] See *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, pp. 64–65 (1932).

The Society was represented at the International Soil Conference held in Cambridge, Mass., June, 1936, by C. A. Hogentogler and W. S. Housel. These representatives submitted to the Executive Committee a full report of the deliberations of the conference, which has been made available to the newly organized Committee D-18 on Soils for Engineering Purposes.

The Society was represented at the International Congress on Building of Bridges and Structures Above Ground held in Berlin, October 1 to 8, 1936, by E. E. Howard. An interesting report from Mr. Howard was published in the December, 1936, *Bulletin.*

The Society will be informally represented at the Second World Petroleum Congress to be held in Paris this June by C. B. Veal, Manager, Research Department, Society of Automotive Engineers.

On invitation from the British Institution of Mechanical Engineers the Society, in conjunction with other American technical societies, has been helpful in suggesting American authors for papers to be submitted at a General Discussion on Lubrication and Lubricants to be held in London next October under the auspices of the Institution.

Various appointments to administrative committees of the Society, to joint committees and committees of other organizations have been made by the Executive Committee and reported currently in the Society *Bulletin.*

Respectfully submitted on behalf of the Executive Committee,

A. C. FIELDNER,
President.

C. L. WARWICK,
Secretary-Treasurer.

June, 1937

REPORT OF COMMITTEE A-1
ON
STEEL

In addition to two series of meetings of Committee A-1 on Steel, the first at Atlantic City, N. J., June 29 and 30, 1936, the second at Chicago, Ill., March 1 to 3, 1937, several special subcommittee meetings have been held during the year to discuss various problems. Satisfactory progress has been made in the numerous activities currently under way. The committee is enabled to propose seven new tentative standards and the adoption as standard of six existing tentative specifications.

In the portion of this report devoted to Activities of Subcommittees are mentioned several important specification projects on which the committee expects to make definite recommendations during the coming year.

Since the 1936 annual meeting, 7 new members have been elected and 3 resignations accepted, the committee membership now totaling 219, of whom 121 are classified as non-producers and 98 as producers; of the former, 92 are consumer and 29 general interest members.

Subcommittee III on Structural Steel for Ships has elected Captain William McEntee, U. S. Navy, Bureau of Construction and Repair, as chairman, this office having been made vacant by the retirement of Rear-Admiral R. M. Watt, also of this Bureau.

Because of a change in position, H. W. Faus, New York Central System, has resigned his affiliation with Committee A-1 and several subcommittees, also as vice-chairman of the committee and chairman of Subcommittee IV on Spring Steel and Steel Springs. He had held these offices respectively from 1932 and 1927. Committee A-1 records its appreciation of his many services in its behalf.

Since the last annual meeting, the committee has lost by death several members, three of whom, R. H. Jeffers, E. T. Ickes and J. C. Ramage, had participated in the work for many years.

Tentative Standards Approved During the Past Year.—Subsequent to the 1936 annual meeting, Committee A-1 on Steel presented to the Society on August 26, 1936, through Committee E-10 on Standards the recommendations listed below, all of which were accepted.[1]

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee A-1 reported the following results of the letter ballot vote, of a total of 136 ballots returned from a committee membership of 215: A 192 – 36 T, affirmative 78, negative none, and 58 ballots marked "not voting;" A 193 – 36 T, affirmative 76, negative 1, and 59 ballots marked "not voting;" A 194 – 36 T, affirmative 75, negative 1, and 60 ballots marked "not voting;" A 195 – 36 T, affirmative 75, negative 1, and 60 ballots marked "not voting."

Tentative Specifications for:

Seamless Steel Boiler Tubes for High-Pressure Service (A 192 - 36 T),
Alloy-Steel Bolting Materials for High Pressure and Temperatures up to 1100 F. (A 193 - 36 T),
Nuts Used in Bolting for High-Pressure Service at Temperatures up to 1100 F. (A 194 - 36 T), and
High-Strength Structural Rivet Steel (A 195 - 36 T).

The new tentative specifications appear in the 1936 *Proceedings*.[1]

Approval of A.S.T.M. Standards by American Standards Association.—During the year, four standard specifications under the jurisdiction of Committee A-1 have been approved as American Standard by the American Standards Association, as follows:

Standard Specifications for Steel for Bridges (A 7 - 36) (A.S.A. G18—1936),
Standard Specifications for Steel for Buildings (A 9 - 36) (A.S.A. G19—1936),
Standard Specifications for Mild Steel Plates (A 10 - 36) (A.S.A. G20—1936),
Standard Specifications for Structural Rivet Steel (A 141 - 36) (A.S.A. G21—1936).

RECOMMENDATIONS AFFECTING STANDARDS

The recommendations of the committee affecting its standard and tentative specifications are listed first in summarized form for convenience; an analysis of the letter ballot on each item is given in Table I. Where desirable, these recommendations are referred to further, being grouped for convenience in the order of the subcommittees responsible for them.

I. Proposed Tentative Standards.—The committee recommends that the following proposed tentative specifications be accepted for publication as tentative as appended hereto:

Proposed Tentative Specifications for:

Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service,[2]
Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes,[3]
Carbon-Silicon-Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels,[4]
Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels,[5]
Low-Carbon-Nickel Steel Plates for Boilers and Other Pressure Vessels,[6]
Molybdenum-Steel Plates for Boilers and Other Pressure Vessels,[7] and
Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods).[8]

II. Proposed Tentative Revisions of Standards.—The committee recommends that revisions, as appended hereto,[9] of the three standard specifications listed below be published as tentative for one year before they are referred to letter ballot of the Society for adoption as standard:

Standard Specifications for:

Structural Rivet Steel (A 141 - 36),

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 606, 627, 634, and 567 (1936); also 1936 Book of A.S.T.M. Tentative Standards, pp. 76, 108, 115, and 7.

[2] See p. 608.

[3] See p. 601.

[4] See p. 565.

[5] See p. 572.

[6] See p. 577.

[7] See p. 582.

[8] See p. 589.

[9] See pp. 1303 to 1305.

High Tensile Strength Carbon-Steel Plates for Pressure Vessels (Plates 2 in. and Under in Thickness) (A 149 – 36), and

High Tensile Strength Carbon-Steel Plates for Fusion-Welded Pressure Vessels (Plates over 2 in. to 4 in., Inclusive, in Thickness) (A 150 – 36).

III. Adoption of Tentative Standards as Standard.—The committee recommends that the following tentative standards be referred to letter ballot of the Society for adoption, as standard:

Tentative Specifications for:

Fabricated Steel Bar or Rod Mats for Concrete Reinforcement (A 184 – 36 T),[1]

Welded Steel Wire Fabric for Concrete Reinforcement (A 185 – 36 T),[1]

Forged or Rolled Steel Pipe Flanges for General Service (A 181 – 35 T),[2]

Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 35 T),[2]

Seamless Low-Carbon-Steel Still Tubes for Refinery Service (A 161 – 35 T),[2] and

Seamless Cold-Drawn Steel Heat-Exchanger and Condenser Tubes (A 179 – 35 T).[2]

IV. Withdrawal of Standards.—The withdrawal of the List of Specifications for Steel Suitable for Fusion Welding (A 151 – 35)[3] is recommended.

The recommendations appearing in this report have been referred to letter ballot of the committee which consists of 219 members, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Certain important features of the committee's work are discussed below, grouped in the order of the respective subcommittees responsible.

Subcommittee II on Structural Steel for Bridges, Buildings and Rolling Stock (F. H. Frankland, chairman).—In order to bring up to date the table of permissible variations in diameter and to make these requirements conform to corresponding tables in other specifications, tentative revisions, appended hereto,[4] are proposed by Subcommittee II in the Standard Specifications for Structural Rivet Steel (A 141 – 36). Revisions of the finish clause in the Tentative Specifications for Structural Nickel Steel (A 8 – 36 T) are being studied in order to bring this clause in line with corresponding clauses in other specifications covering structural steel.

Subcommittee IX on Steel Tubing and Pipe (T. G. Stitt, chairman).—Because of the increasing use of intermediate, or low-alloy, seamless steel still tubes and seamless steel heat-exchanger and condenser tubes, proposed tentative specifications covering these materials are recommended, as appended hereto.[5] These are companion specifications to previous specifications prepared by the subcommittee and issued by the Society and they have been approved by the special section comprising representatives of the American Petroleum Institute and Subcommittee IX.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 571, 575 (1936); also 1936 Book of A.S.T.M. Tentative Standards, pp. 19, 23.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, pp. 575, 579, 590, 585 (1935); also 1936 Book of A.S.T.M. Tentative Standards, pp. 82, 65, 101, 71.

[3] 1936 Book of A.S.T.M. Standards, Part I, p. 93.

[4] See p. 1305.

[5] See pp. 601 and 608.

Since no adverse criticism has been received, the four tentative specifications covering various pipe and tubing material, as listed previously in the report, are proposed for adoption as standard.

Important and far-reaching changes are being developed involving methods for designating wall thickness as provided in the Standard Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 36) and Tentative Specifications for Seamless Steel Boiler Tubes for High-Pressure Service (A 192 – 36 T). These changes embody the substitution of decimals in the place of B.w.g. or fractions. New tables covering standard weights and permissible variations are being developed and are expected to be recommended for publication as tentative.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. PROPOSED TENTATIVE STANDARDS			
Specifications for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service	55	0	80
Specifications for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes	58	0	77
Specifications for Carbon-Silicon-Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels	73	1	61
Specifications for Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels	70	1	64
Specifications for Low-Carbon-Nickel Steel Plates for Boilers and Other Pressure Vessels	74	1	60
Specifications for Molybdenum-Steel Plates for Boilers and Other Pressure Vessels	72	1	62
Specifications for Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods)	69	5	61
II. PROPOSED TENTATIVE REVISIONS OF STANDARDS			
Specifications for Structural Rivet Steel (A 141 – 36)	85	0	50
Specifications for High Tensile Strength Carbon-Steel Plates for Pressure Vessels (Plates 2 in. and Under in Thickness) (A 149 – 36)	81	0	54
Specifications for High Tensile Strength Carbon-Steel Plates for Fusion-Welded Pressure Vessels (Plates over 2 in. to 4 in., Inclusive, in Thickness) (A 150 – 36)	77	0	58
III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Specifications for Fabricated Steel Bar or Rod Mats for Concrete Reinforcement (A 184 – 36 T)	81	0	54
Specifications for Welded Steel Wire Fabric for Concrete Reinforcement (A 185 – 36 T)	71	0	64
Specifications for Forged or Rolled Steel Pipe Flanges for General Service (A 181 – 35 T)	74	0	61
Specifications for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 35 T)	61	5	69
Specifications for Seamless Low-Carbon-Steel Still Tubes for Refinery Service (A 161 – 35 T)	54	1	80
Specifications for Cold-Drawn-Steel Heat-Exchanger and Condenser Tubes (A 179 – 35 T)	58	1	76
IV. WITHDRAWAL OF STANDARD			
List of Specifications for Steel Suitable for Fusion Welding (A 151 – 35)	89	5	41

Two sections of Subcommittee IX are preparing specifications covering respectively electric-fusion spiral butt-welded pipe and alloy-steel boiler and super-heater tubes. The demand for these materials is such that there is definite need for standardized specification requirements which will be met by the issuance of A.S.T.M. tentative standards.

Subcommittee XI on Boiler Steel (E. J. Edwards, chairman).—In order to bring the requirements for firebox steel in the Standard Specifications for Boiler and Firebox Steel for Locomotives (A 30 – 33) in line with the latest requirements of the Association of American Railroads, the subcommittee plans to recommend revisions in the A.S.T.M. specifications.[1]

This subcommittee has received various comments, including proposals from the A.S.M.E. Boiler Code Committee, concerning the Standard Specifications for High Tensile Strength Carbon-Steel Plates for Pressure Vessels (Plates 2 in. and Under in Thickness) (A 149 – 36) and the Standard Specifications for High Tensile Strength Carbon-Steel Plates for Fusion-Welded Pressure Vessels (Plates over 2 in. to 4 in., Inclusive, in Thickness) (A 150 – 36). At the same time requests have been received from various sources including the A.S.M.E. Boiler Code Committee for specification requirements covering various classes of alloy-steel plates for boilers and other pressure vessels. Careful study has been made of the entire matter by a special section and as a result of this work tentative revisions are proposed in the two existing standard specifications and in addition four new specifications as listed previously in this report are recommended for publication as tentative. The subcommittee plans to review carefully any comments received concerning the proposed new specifications and has in mind that any essential revisions can be made while the specifications are tentative.

Subcommittee XXI on Steel for Welding (N. L. Mochel, chairman).—Because the specifications itemized in the List of Specifications for Steel Suitable for Fusion Welding (A 151 – 35) are not considered to cover adequately requirements for welding in view of more recent developments in the art, the withdrawal of the list is recommended. In recommending its withdrawal, the subcommittee suggests that the existing specifications covering various types of materials offered for fusion welding be revised to incorporate specific requirements for welding or to include a statement that the material is suitable for welding.

As the result of several meetings, and with the close cooperation of representatives of the American Welding Society, the subcommittee is enabled to recommend for publication proposed Tentative Specifications for Iron and Steel Filler Metal, (Arc-Welding Electrodes and Gas-Welding Rods), as appended hereto.[2] Various specification requirements incorporated have been discussed at length and it is believed their issuance as an A.S.T.M. tentative standard would be of definite service in meeting the need for standardized requirements for the materials.

Subcommittee XXII on Valves, Fittings, Piping and Flanges for High-Temperature Service (A. E. White, chairman).—This subcommittee is developing extensive revisions in the Tentative Specifications for Alloy-Steel Bolting Materials for High Pressure and Temperatures to 1100 F. (A 193 – 36 T) and it is expected these revisions will be recommended to the Society for approval during the coming year.[1]

Because of the rather widespread use of the carbon-molybdenum alloy (ferritic steel P1) covered in the Standard Specifications for Seamless

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[1] Subsequent to the annual meeting Committee A-1 presented these revisions as tentative to the Society through Committee E-10 on Standards, see Editorial Note, p. 86.—Ed.

[2] See p. 589.

Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158-36), the subcommittee plans to recommend a new tentative specification to be prepared covering this alloy composition alone.[1] A detailed study of the standard has indicated the desirability of deleting certain alloy compositions covered with the addition of others for which there is more important demand. The subcommittee expects to recommend these changes and propose that the standard be reverted to the status of tentative.[1]

The committee is considering clarifying the use of the term "stabilized" in certain of the specifications covering various types of alloy steel for service at elevated temperatures.

Subcommittee XXIII on Die Steels.—It has been decided to disband Subcommittee XXIII on Die Steels until sufficient data are available on die casting practices to proceed with standardization work on die steels in common use. This action is in accord with the recommendations of Committee B-6 on Die-Cast Metals and Alloys.

This report has been submitted to letter ballot of the committee which consists of 219 members; 113 members returned their ballots, of whom 105 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

H. H. MORGAN,
Chairman.

H. P. BIGLER,
Secretary.

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[1] See Editorial Note below.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee A-1 on Steel presented to the Society on August 26, 1937, through Committee E-10 on Standards the following recommendations:

Proposed Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F.,

Proposed Tentative Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 – 37 T), which together with the above specifications are a revision of and supersede the Standard Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 – 36),

Proposed Revision of Tentative Specifications for Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures (A 193 – 36 T),

Proposed Tentative Revision of Standard Specifications for Boiler and Firebox Steel for Locomotives (A 30 – 33), and

Withdrawal of Standard Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 – 36).

The above recommendations were accepted by Committee E-10 and appear on pp. 633, 622, 616 and 1303, respectively.

REPORT OF RESEARCH COMMITTEE
ON
YIELD POINT OF STRUCTURAL STEEL

INTRODUCTION

Shortly after appointment in 1926, this committee made a statement of the scope of its work.[1] The essential factors to be considered were:

A. The significance of the yield point to the engineer,
B. The range in the yield point when the material is furnished under specifications, and
C. The testing procedure which will insure sufficiently accurate determination of the yield point with a minimum of expense.

The main efforts of the committee have been spent on items B and C. The committee has limited its tension test programs to bar and standard flat specimens of 8-in. gage length held in the testing machine by serrated wedges. Reports of five series of tests on over 1000 specimens have been made to this Society.[2]

During the depression uncertainty of operation at the steel mills caused cessation in the testing program of the committee. A series of 150 tests, No. 351, on the effect of aging on the yield point and other mechanical properties of structural steel has been finally completed and is reported herein. This report also contains a summary of the work of the committee and certain recommendations.

The test programs of the committee have been made possible through the effective cooperation of the Illinois Steel Co., represented by Mr. John Brunner, and the Bethlehem Steel Co., represented by Mr. E. F. Kenney. Mr. Brunner, until the time of his death, and Mr. F. S. Crane, his assistant, were always very helpful in planning and executing test programs. The committee has also had the cooperation of the laboratories of these two steel companies and the materials testing laboratories at Rensselaer Polytechnic Institute and at The University of Wisconsin. All materials and testing services were generously contributed by the companies and institutions named, for which the committee here desires to express its cordial thanks.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I, p. 646 (1927).

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part I, p. 105 (1928); Vol. 29, Part I, p. 101 (1929); Vol. 30, Part I, p. 133 (1930); Vol. 31, Part I, p. 136 (1931).

TESTS OF SERIES NO. 351

Purpose.—The purpose of these tests was to determine the effect of aging on the yield point of structural steel rods.

Material.—The material for the tests conformed to the Standard Specifications for Steel for Bridges (A 7 – 34) of the American Society for Testing Materials.[1] The billets were selected and the rods rolled in the presence of a representative of the committee acting in cooperation with a representative of the Illinois Steel Co. at Gary, Ind., in the presence of another representative of the committee acting with a representative of the Bethlehem Steel Co. at Bethlehem, Pa., during the period November 19 to December 17, 1935.

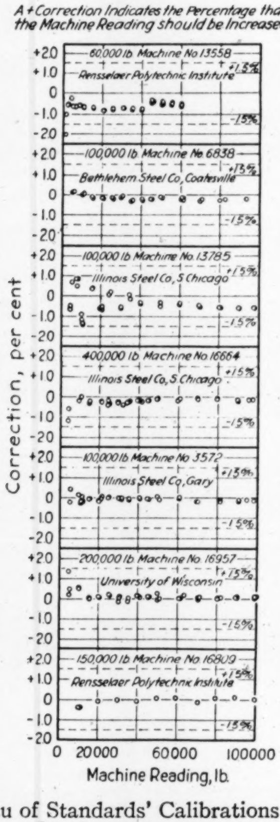

FIG. 1.—Results of Bureau of Standards' Calibrations of Testing Machines, Made October 19 to 26, 1931.

At each mill three 20-ft. lengths were selected from each size of rod from the mid-portion of the bloom. Beginning at the end of the selected length nearest the top of the ingot, ten portions 18-in. long were marked

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[1] 1934 Supplement to Book of A.S.T.M. Standards, p. 1.

1A, 1B, 1C, 1D, etc. Specimens from the second length were marked 2A, 2B, etc.; those from the third length were marked 3A, 3B, etc. The remaining 5-ft. portions were held in reserve until the tests were completed.

The A and J specimens were tested at the mill when 1 day old. Specimens marked B, D, F, and I were also tested at the mill at ages of 7, 28, 180 and 360 days, respectively. Specimens marked C, E, G, and H were shipped to the cooperating laboratories and tested at corresponding ages.

TABLE I.—MILL TESTS ON SPECIMENS FOR SERIES NO. 351.

Mill	Diameter of Bar, in.	Temperature at Last Pass, deg. Fahr.	Chemical Analyses					Tensile Strength,[a] lb. per sq. in.	Yield Point, lb. per sq. in.	Elongation in 8 in., per cent	Heat Number
			Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Copper, per cent				
Illinois Steel Co., Gary...	$\frac{7}{8}$	1872	0.24	0.42	0.015	0.032	....	65 270	42 730	29.25	88563
	$1\frac{1}{4}$	1908	0.23	0.44	0.013	0.029	....	63 050	39 510	33.50	83467
Bethlehem Steel Co....	$\frac{7}{8}$	1680	0.17	0.62	0.031	0.044	0.30	not made	not made	....	45E393
	1	1773	0.16	0.62	0.032	0.043	0.28	not made	not made	....	45E393
	$1\frac{1}{4}$	1670	0.18	0.63	0.030	0.043	0.30	not made	not made	....	45E39

[a] Speed at yield point, 0.5 in. per min.; at tensile strength, 2.0 in. per min.

TABLE II.—MACHINES AND SPEED USED IN TESTS OF SERIES NO. 351.
Includes Tests at all Ages

Laboratory	Testing Machine	Diameter of Bar, in.	Speed of Head of Testing Machine, in. per min.			Number Assisting in Tests	Time in Minutes and Seconds		
			to Yield Point	at Yield Point	to Tensile Strength		Zero Load to Yield Point	Zero Load to Tensile Strength	Total per Test
Illinois Steel Co., Gary..........	Riehle 300,000 lb.	$\frac{7}{8}$	0.5	0.175	2.0	3	1:51	4:21	6:26
		$1\frac{1}{4}$	0.5	0.175	2.0	3	1:58	4:31	6:52
University of Wisconsin.........	Riehle 200,000 lb.	$\frac{7}{8}$	0.64	0.184	1.25	2	1:38	5:24	11:00
		$1\frac{1}{4}$	0.64	0.184	1.25	2	1:51	6:06	12:25
Lehigh University..............	Olsen 300,000 lb.	$\frac{7}{8}$	0.85	0.175	0.85	3	....	6:35	9:48
		1	0.85	0.175	0.85	3	....	6:46	9:48
		$1\frac{1}{4}$	0.85	0.175	0.85	3	....	7:45	10:24
Rensselaer Polytechnic Inst......	Riehle 150,000 lb.	$\frac{7}{8}$	1.0	0.25	1.20	3	....	4:37	6:45
		1	1.0	0.25	1.20	3	....	5:01	6:15
		$1\frac{1}{4}$	1.0	0.25	1.20	3	....	5:38	7:00

The University of Wisconsin laboratory cooperated with the Gary laboratory of the Illinois Steel Co. and the Rensselaer Polytechnic Institute laboratory cooperated with the Bethlehem Steel Co. laboratory.

Preparation of Specimens.—Specimens for the regular mill tests were selected and prepared in accordance with the practice adopted at the mill. The test specimens were not separated from the parent length until the day scheduled for test.

TABLE III.—STRENGTH AND DUCTILITY DATA FOR TESTS OF SERIES NO. 351.

Each average represents 3 tests, except one day averages represent 6 tests.

Laboratory	Diameter of Bar, in.	Age at Test, days	Yield Point, lb. per sq. in.	Average Deviation of Yield Point from Mean, per cent	Tensile Strength, lb. per sq. in.	Reduction of Area, per cent	Elongation in 8 in., per cent	Percentage Change from 1-day Value	
								Yield Point	Elongation
Illinois Steel Co., Gary.....	$\frac{7}{8}$	1	40 810	0.5	64 125	48.50	29.38	.....	.....
		7	39 400	0.2	64 600	56.88	28.83	−3.5	−1.9
		28	39 420	0.7	65 170	62.28	31.83	−3.4	+8.3
		180	39 240	0.7	64 710	63.01	30.67	−3.8	+4.4
		360	38 780	0.7	65 760	62.58	31.08	−5.0	+5.8
	$1\frac{1}{4}$	1	38 990	1.2	63 860	49.97	30.71	.....	.....
		7	38 330	0.7	64 030	55.68	33.33	−1.7	+8.5
		28	37 050	0.7	64 170	58.48	32.50	−5.0	+5.8
		180	37 540	0.6	64 710	58.41	33.00	−3.7	+7.5
		360	37 240	0.4	65 160	58.25	32.83	−4.5	+6.9
	$\frac{7}{8}$	½	40 520	0.4	62 260	34.64	24.58	−3.1	−6.6
		1	41 800	0.3	62 550	47.53	26.33	.....	.....
	$1\frac{1}{4}$	½	37 930	1.8	63 010	49.13	31.33	.....	.....
University of Wisconsin....	$\frac{7}{8}$	7	40 640	1.6	62 780	49.69	29.92	.....	.....
		28	41 410	1.8	64 960	56.26	29.63	.....	.....
		180	39 110	0.9	62 520	60.50	31.79	.....	.....
		360	39 850	3.2	62 160	59.30	30.38	.....	.....
	$1\frac{1}{4}$	7	37 760	1.2	63 340	55.40	31.33	.....	.....
		28	37 180	0.1	63 830	59.03	32.25	.....	.....
		180	37 020	1.4	63 500	59.50	32.38	.....	.....
		360	38 010	0.5	63 900	59.20	33.50	.....	.....
Lehigh University..........	$\frac{7}{8}$	1	42 320	0.7	61 900	52.29	30.34	.....	.....
		7	42 340	1.3	62 160	56.00	31.04	+0.0	+2.3
		28	42 530	0.5	61 790	62.86	33.00	+0.5	+8.8
		180	42 000	0.4	62 160	65.40	31.08	−0.8	+2.4
		360	41 860	0.3	62 440	65.50	31.25	−1.1	+3.0
	1	1	40 220	1.0	60 945	50.27	30.38	.....	.....
		7	40 470	1.7	61 310	55.32	32.38	+0.6	+6.6
		28	39 600	1.4	60 330	59.60	31.83	−1.5	+4.8
		180	39 710	1.3	61 310	65.50	32.33	−1.3	+6.4
		360	39 590	1.9	61 270	65.90	32.42	−1.6	+6.7
	$1\frac{1}{4}$	1	39 885	1.3	60 390	45.40	29.74	.....	.....
		7	39 650	0.6	60 910	45.36	31.25	−0.6	+5.1
		28	39 140	2.8	60 150	55.52	32.79	−1.9	+10.3
		180	39 210	0.6	60 730	64.90	33.42	−1.7	+12.4
		360	38 490	0.5	60 790	65.20	35.00	−3.5	+17.7
Rensselaer Polytechnic Inst..................	$\frac{7}{8}$	7	42 100	0.9	61 870	54.54	29.38	.....	.....
		28	42 380	0.8	62 190	60.89	32.25	.....	.....
		180	41 850	0.2	62 680	65.10	33.00	.....	.....
		360	42 340	0.4	62 540	64.70	32.59	.....	.....
	1	7	39 840	1.3	61 010	55.12	30.46	.....	.....
		28	40 220	0.7	61 610	61.32	33.00	.....	.....
		180	38 740	0.9	61 080	65.66	33.58	.....	.....
		360	39 980	1.4	61 710	64.70	33.67	.....	.....
	$1\frac{1}{4}$	7	39 700	0.6	60 350	47.44	30.42	.....	.....
		28	39 400	1.2	60 720	56.73	34.92	.....	.....
		180	38 300	0.6	60 720	64.20	35.08	.....	.....
		360	39 560	1.4	61 090	64.00	34.83	.....	.....
Average for all laboratories.	$\frac{7}{8}$	1	41 565	...	63 015	50.40	29.86	.....	.....
		7	41 120	...	62 855	54.28	29.79	.....	.....
		28	41 435	...	63 530	60.58	31.68	.....	.....
		180	40 550	...	63 020	63.50	31.64	.....	.....
		360	40 710	...	63 225	63.02	31.33	.....	.....
	1	1	40 220	...	60 945	50.27	30.38	.....	.....
		7	40 155	...	61 160	55.22	31.42	.....	.....
		28	39 910	...	60 970	60.46	32.42	.....	.....
		180	39 225	...	61 195	65.58	32.95	.....	.....
		360	39 785	...	61 490	65.30	33.05	.....	.....

Table III.—(*Continued*).

Laboratory	Diameter of Bar, in.	Age at Test, days	Yield Point, lb. per sq. in.	Average Deviation of Yield Point from Mean, per cent	Tensile Strength, lb. per sq. in.	Reduction of Area, per cent	Elongation in 8 in., per cent	Percentage Change from 1-day Value	
								Yield Point	Elongation
Average for all laboratories (*Continued*)	1¼	1	39 440	...	62 125	47.69	30.23	.....	.....
		7	38 860	...	62 160	50.97	31.59	.....	.....
		28	38 195	...	62 220	57.45	33.12	.....	.....
		180	38 020	...	62 415	61.76	33.47	.....	.....
		360	38 325	...	62 735	61.67	34.05	.....	.....
Average of all bars.........	......	1	40 445	...	62 245	49.29	30.11	.....	.....
		7	40 025	...	62 235	53.14	30.83	.....	.....
		28	39 835	...	62 490	59.30	32.40	.....	.....
		180	39 270	...	62 410	63.22	32.63	.....	.....
		360	39 570	...	62 680	62.93	32.76	.....	.....
Illinois Steel Co., Gary, Average of all bars.....	......	1	39 900	2.3	63 995	49.24	30.05	.....	.....
		7	38 865	1.4	64 315	56.28	31.08	−2.6	+3.4
		28	38 235	3.1	64 670	60.38	32.17	−4.2	+7.1
		180	38 390	2.2	64 710	60.71	31.84	−3.8	+6.0
		360	38 010	2.0	65 460	60.42	31.96	−4.7	+6.4
Lehigh University, average of all bars............	......	1	40 810	2.6	61 080	49.32	30.15	.....	.....
		7	40 820	2.7	61 460	52.23	31.56	+0.0	+4.7
		28	40 425	3.7	60 755	59.33	32.54	−0.9	+7.9
		180	40 305	2.9	61 400	65.3	32.28	−1.2	+7.1
		360	39 980	3.5	61 500	65.5	32.89	−2.0	+9.1

Method of Testing.—Tests made at the University of Wisconsin and at Rensselaer Polytechnic Institute were run on the machines calibrated by the National Bureau of Standards' representatives in 1931, see Fig. 1. All machines were calibrated during the aging tests by approved methods. Specimens were punch-marked over the entire length at 1-in. intervals. Dimensions of cross-section were measured and recorded to the nearest 0.001 in.

Specimens were loaded to three-fourths of the estimated yield point with any controllable speed, then the rate was reduced to 0.15 to 0.25 in. per min. until the yield point was obtained by drop of the beam. The speed was then increased to 1 to 2 in. per min. and the maximum load determined. The regular mill tests were run at the customary speeds.

At each mill the visiting representative of the committee witnessed the 1-day tests. Owing to lack of sufficiently low speed regulation on the testing machine at the Bethlehem mill laboratory, all tests scheduled for that laboratory were made at Lehigh University.

Results of Tests.—The data on composition, finishing temperature and mill tests are given in Table I. Information on the types of testing machines used and the average time required in performing tests is recorded in Table II. The principal data obtained from the tests are recorded in Table III and the influence of age on mechanical properties is shown in Figs. 2, 3, 4, and 5.

The tests indicate that the effect of age on yield point and tensile strength of hot-rolled structural steel of the grade herein used is small.

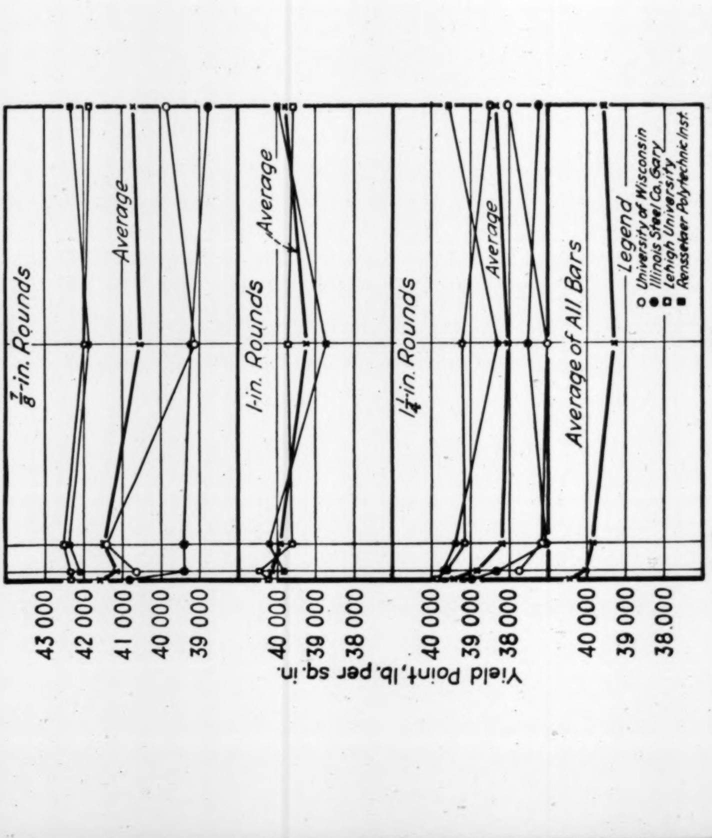

FIG. 2.—Influence of Age on Yield Point of Rods.

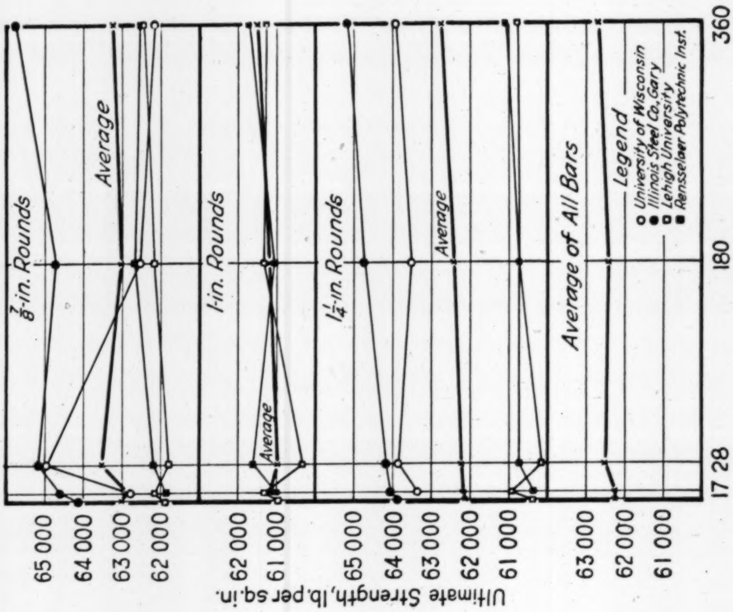

FIG. 3.—Influence of Age on Ultimate Strength of Rods.

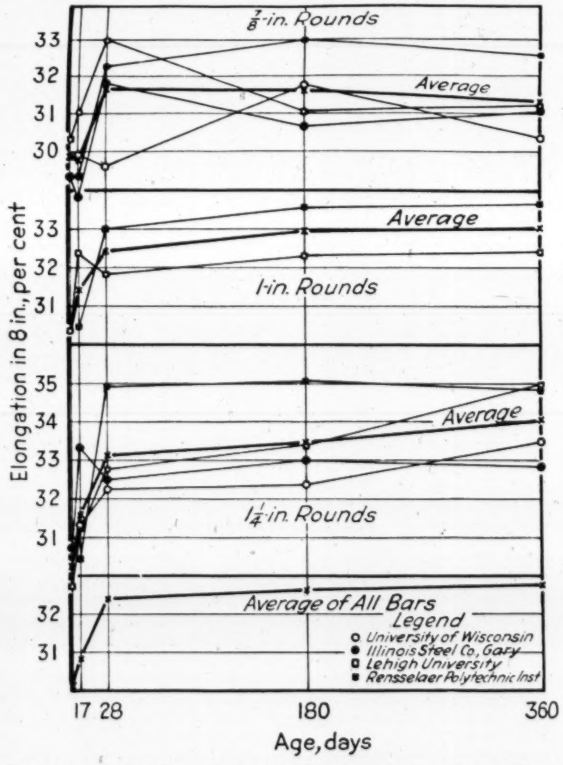

FIG. 4.—Influence of Age on Elongation in 8 in. of Rods.

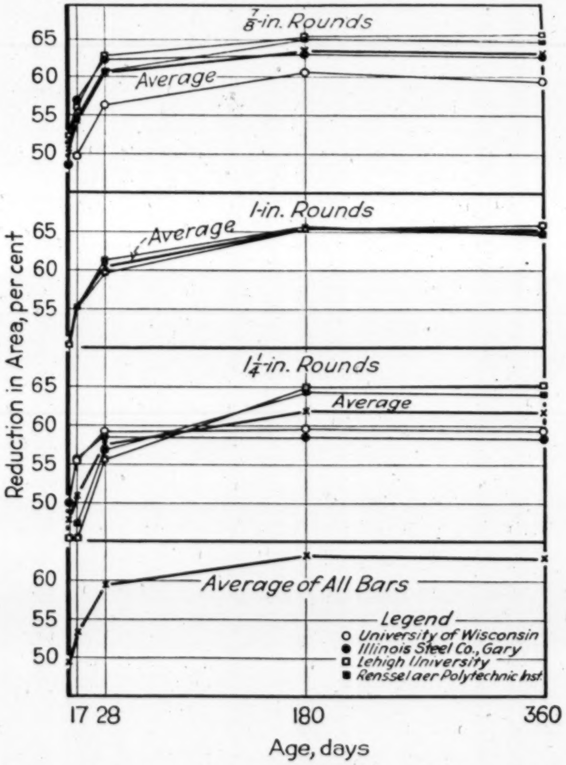

FIG. 5.—Influence of Age on Reduction of Area of Rods.

Considering the average results for all laboratories, the yield point showed an average reduction of approximately 2 per cent between 1 day and 1 month with little or no change thereafter; on the other hand the tensile strength increased slightly. By comparing age relationships for the tests conducted at Gary and at Lehigh, the only laboratories making 1-day tests, variables due to differences in testing machines are eliminated. Such comparisons indicate somewhat larger reductions in yield point than do the averages for all laboratories. The reduction in yield point between 1 day and 1 yr. for the data obtained at Gary averaged 4.7 per cent and for the data obtained at Lehigh the reduction averaged 3.7 per cent.

There is a material improvement in ductility as evidenced by an average increase of 30 per cent in reduction of area and 8 per cent in percentage elongation in 8 in. between ages of 1 day and 180 days. Most of this increase in ductility took place within the first month. The pronounced increase in ductility between ages of 2 hr. and 1 day as shown by the small number of tests made at Gary is remarkable.

These changes in properties, consistently exhibited in the material from both mills, and their causes appear worthy of more study on the part of the metallurgist. It is conceivable that other grades of structural steel may exhibit greater variations than were encountered in these tests; also that the variations may be considerably greater in thin angles and wide flanged shapes.

RÉSUMÉ OF ANSWERS TO QUESTIONNAIRE DEALING WITH SIGNIFICANCE OF THE YIELD POINT TO THE DESIGNING ENGINEER

In 1933 approximately 80 questionnaires were sent out to representative designing engineers and engineers who were intimately connected with designing involving the use of structural steel. Nineteen replies received from these engineers other than members of this committee form the basis for the following résumé.

Q. 1. What physical changes in structural steel are signified by the yield point?

Fourteen replies indicated that the yield point was considered to be the stress at which permanent deformation began or elastic breakdown of the material commenced. Five made no answer.

Q. 2. Do you consider the yield point in determining the allowable working stress? If so, when and how do you take the yield point into account?

Sixteen replies stated that the engineers considered the yield point or believed that the allowable unit stress should be a proportion of the yield point.

Q. 3. In determining the allowable unit stress, is the yield point of more or less value than: the proportional limit; the elastic limit; the useful limit point?

In 13 replies the yield point was considered the most valuable index of the four mentioned; two considered that the yield point was less valuable than the proportional limit or elastic limit although admitting the greater ease of its determination; and one considered the useful limit point to be the most valuable if properly determined.

Q. 4. What especial significance, if any, does the yield point have with respect to the behavior of: (*a*) a tension member; (*b*) a compression member?

(*a*) Nine replies indicated that the excessive deformation accompanying the stressing beyond the yield point would be objectionable. Six replies indicated it was a very important stress to be considered in designing tension members. One reply indicated that a small amount of stressing beyond the yield point would cause equalizing of stresses in the member in question and in adjoining members.

(*b*) Twelve replies stated that the yield point was the upper limit of column strength. Two replies implied that it was of little importance. One reply indicated that it was sometimes important in estimating column strength.

Q. 5. Does the yield point furnish information which cannot be estimated with reasonable accuracy from the ultimate strength? In what way?

Fourteen replies indicated that the yield point was a better basis for estimating allowable unit stresses than the ultimate. Two replies were to the effect that the yield point could be estimated from the ultimate strength with reasonable accuracy but the engineer would prefer to have knowledge of the yield point.

Q. 6. Do you rely upon mill tests, commercial testing laboratories, or your own laboratory for determinations of the yield point of steel for important structures?

Eight replies indicated that mill tests were relied upon for information on tensile properties. Three replies stated that commercial laboratories were employed for making tests. Two replies indicated that their own laboratories did the work, two stated that mill or commercial laboratories were employed, one reply indicated that all types of laboratories had been used, and one indicated that the mill tests were checked by a commercial laboratory.

Q. 7. In determining the quality of material for important structures, how many tension tests do you require (*a*) from each heat; (*b*) from each heat per type of shape; (*c*) from different locations in a given shape?

Eight replies stated that the A.S.T.M. specifications were followed. Three indicated that there was no regular procedure in these matters. One asserted that the A.R.E.A. specifications were followed. One reply indicated that the number of specimens varied from one per heat to one per structural member, that at least one specimen was taken for each type of member and that only one location in a member was sampled. One reply stated that one test specimen was taken from each heat for each type of shape, and one reply indicated that the testing schedule was varied in circumstances (presumably in accordance with the importance of the work).

Q. 8. Do you favor the use of slower speeds than commonly practiced in the determination of the yield point? (For example, would you favor speeds as slow as 0.1 to 0.2 in. per min. for an 8-in. gage length when the load approaches the yield point?)

Eight replies favored slower speeds, three did not favor slower speeds, two replies indicated that the authors believed the speed should be slow enough to enable the operator to keep the scale beam in balance and determine accurately the drop of beam and one indicated that constant speeds should be used to avoid variations in results.

Q. 9. Do you favor basing the yield point determination on (*a*) drop of beam; (*b*) a given strain; (*c*) a given permanent set?

Thirteen replies favored the use of drop of beam, two indicated that the dividers were used to check the drop of beam, and one stated that the yield point should be determined from plotted stress-deformation curves.

Q. 10. Have you any other suggestions for improving the procedure you have mentioned in replies to Q. 6 and Q. 7?

Two reaffirmed their opinion that slower speed should be used in testing, one believed that calibration of testing machines should be required and that the reporting of speeds used in testing should be required. One believed that specimens should be taken from the thickest and thinnest parts of sections rolled, and one indicated that the drop of beam determinations should be checked by dividers. One reply stated that the author believed that satisfactory material could be obtained by demanding from the fabricating company a written guarantee to the effect that the material used came from heats reported in mill tests.

CONCLUSIONS FROM THE WORK OF THE COMMITTEE

1. Designers of structural steel bridges and buildings attach much significance to the yield point in arriving at allowable working stresses both for tension and compression members, and prefer that the yield point shall be determined at speeds sufficiently slow to insure reliable data.

2. The yield point in tension is a reliable index of the yield point in compression for hot-rolled structural steel (Series No. 261, 1928 report).[1]

3. It is not possible to predict accurately the yield point from the ultimate strength, since the ratio of yield point to tensile strength varied from 0.49 to 0.64 for the whole shapes tested and only about half of the shapes had ratios of yield point to tensile strength within 5 per cent of the mean ratio (see Fig. 5, Series Nos. 271 and 281, 1930 report).[2]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part I, p. 105 (1928).

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 133 (1930).

4. The gagging of girder beams and H-columns produced a slight increase in yield point which from a practical standpoint is negligible (Series No. 281).[1] Other tensile properties were unaffected. So far as these tests indicated, contact of the member with the cooling skids had little effect on tensile properties.

5. The following were the best locations for a single specimen representing the given structural shape: for plates and angles, the intermediate location; for channels and I-beams, the center of the web; for girder beams and H-beams, points midway between toe and root of flange (see Series Nos. 271 and 281, 1930 report).[1]

6. The range in yield point of different portions of shapes was 29,090 to 38,880 lb. per sq. in., whereas the variation in tensile strength, 52,520 to 62,400 lb. per sq. in., was proportionately much less (Series Nos. 271 and 281).[1] In most cases this variation in yield point appeared to be due to differences in cooling and rolling to which the different portions of the shapes were subjected. In six out of eight pairs of shapes tested the yield points of the light shapes exceeded the yield points of the heavy shapes by 11 to 19 per cent; in only two pairs of shapes did the yield points of the heavy shapes exceed those of the light shapes and in one case the discrepancy was apparently due to difference in composition.

7. Aging reduces the yield point by a small amount, in these tests less than 5 per cent, and slightly increases the tensile strength of hot-rolled rods of low-carbon steel. Aging does effect a material increase in the ductility of such material during the first month after rolling.

8. Of the four methods used to detect yield point, the multiplying dividers method was slightly more reliable than the drop-of-beam method. The 2-in. strain gage was not quite so satisfactory as the dividers with an 8-in. gage, and scaling was the least satisfactory index of the yield point of the methods tried. All things considered, the drop-of-beam method being simplest and quickest appeared to be sufficiently reliable. In using the drop-of-beam method at speeds of 0.5 in. per min., or less, care should be exercised to avoid false indications of yield point due to the momentary drop which accompanies overstrain at the grips.

9. A large part of the work of the committee has dealt with the effect of pulling speed on the yield point. For the $\frac{3}{4}$-in. round rods of carbon, silicon, and nickel structural steels (Series No. 261),[1] raising the free speed of head from 0.05 in. per min. to 2 in. per min. increased the average yield point 9 per cent for the carbon steel, 7 per cent for the nickel steel, and 12 per cent for the silicon steel bars. At a speed of 0.2 in. per min. the yield point by drop of beam averaged approximately 600 to 1000 lb. per sq. in. higher than at a speed of 0.05 in. per min.

For these same bar steels, the percentage range in the average yield points obtained at the five different laboratories varied from 2 to 5 per cent for the speeds of $\frac{1}{8}$ to $\frac{1}{2}$ in. per min. The maximum ranges in individual test values was approximately double these percentages.

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[1] *Loc. cit.*

Considering the entire range of speeds and averages of five specimens, the maximum range in drop-of-beam yield points was 10 per cent for the carbon steel, 6 per cent for the nickel steel and 11 per cent for the silicon steel rods.

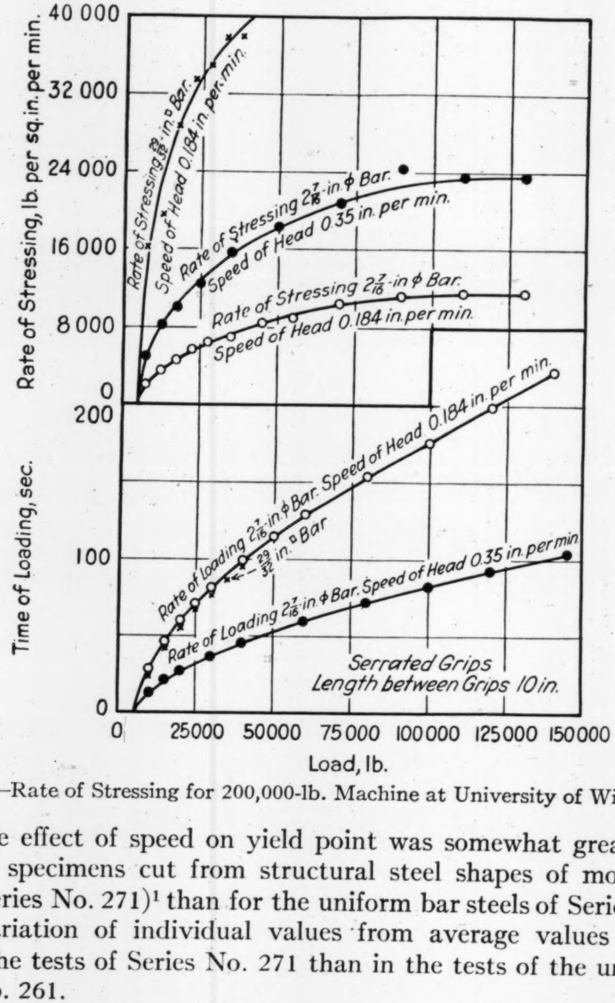

FIG. 6.—Rate of Stressing for 200,000-lb. Machine at University of Wisconsin.

10. The effect of speed on yield point was somewhat greater for the rectangular specimens cut from structural steel shapes of more variable material (Series No. 271)[1] than for the uniform bar steels of Series No. 261,[1] and the variation of individual values from average values was much greater in the tests of Series No. 271 than in the tests of the uniform bars of Series No. 261.

11. The total time of testing was not materially affected by reducing momentarily the speed of machine in the vicinity of yield point to 0.2 in. per min.

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[1] *Loc. cit.*

12. The relation of the yield point in tension by drop of beam to free speed of movable head was approximately the same for the screw-gear-driven lever machines and the Amsler hydraulic machine, used in Series No. 282.[1] The shapes of the yield point – speed-of-head curves for tests made by the two types of machine were found to be substantially the same.

13. In the progress report of 1931 (Series No. 291)[2] it was shown that the ratio of rate of strain to speed of head was practically constant for a given machine for the speeds and loads used and that the ratio varied considerably for the different machines, ranging from approximately $\frac{1}{16}$ to $\frac{1}{50}$ in. per min. for the grips and ranges of loading used. Further study of the rate of strain at different loads on the 200,000-lb. machine at the University of Wisconsin, using serrated grips, Fig. 6, indicates that the ratio of rate of strain to speed of head increases rapidly with the load in the lower load ranges and much less rapidly above one-fourth machine capacity.

In the 1931 report, a simple method for calibrating the speed-rate of strain relationship for any machine was indicated.

14. On the basis of representative specimens, it was noted in the 1930 report that the average yield point for a given shape by mill tests exceeded the corresponding value from cooperating laboratory tests by 3 to 17 per cent and that the average tensile strength by mill tests exceeded the corresponding tensile strength from cooperating laboratory tests from 3 to 12 per cent. It did not appear that these discrepancies could be due to differences in speed. Comparisons made on the basis of average weighted mean values for the whole shapes made the discrepancies between mill and cooperating laboratory tests still greater (see Table XVI, 1930 report).

The results of the National Bureau of Standards' calibrations, Fig. 1, indicate that none of the machines had errors in excess of 1.5 per cent in the load ranges which were significant in the tests and in most cases the machines were accurate within 1 per cent. Hence, discrepancies between mill and cooperating laboratory tests cannot be ascribed to errors in the machines used.

RECOMMENDATIONS

As a result of its studies, the committee makes the following recommendations for the consideration of Committee A-1 on Steel and Committee E-1 on Methods of Testing, and others interested.

1. Since the yield point of structural steel is of sufficient importance to the designing engineer to warrant the practice of increased accuracy in the determination of the yield point, present methods of procedure should be improved to attain that end. It is therefore recommended that the speed requirement for the determination of the yield point based on the

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part I, p. 101 (1929).
[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 136 (1931).

speed of head running free be discarded and that in the vicinity of the yield point the rate of pulling shall be limited by rates of strain or by rates of stress. Tentatively for wedge grips and an 8-in. gage length the suggested maximum limit for the rate of strain in the vicinity of the yield point is 0.0015 in. per in. per min., which would correspond to a stress limit of 45,000 lb. per sq. in. per min.[1]

2. Machines should be accurate within 1 per cent in the load range at which the yield point and ultimate strength are determined.

3. The drop of beam or halt in motion of the pointer on the dial are sufficiently accurate methods of determining the yield point in practically all cases. A pair of multiplying dividers with gage length of 8 in. and a 10 to 1 multiplication arranged to indicate an elongation of 0.5 per cent afford a slightly more reliable method of detecting the yield point and are recommended for emergencies.

4. The following locations are recommended for specimens cut from structural steel shapes: for plates and angles, the intermediate position between edge and center of plate and between toe and heel of angle; for channels and I-beams, the center of the web; for H-beams and other wide flange members, the position midway between toe and root of flange.

5. The fundamental reasons for age effects on all steels, the magnitude of age effects on the yield point and ductility of structural steels other than carbon steels, and the magnitude of the age effects on the yield point of steel shapes should be investigated.

6. It is further recommended that a study be made of the factors affecting the yield point of the various new alloy steels entering the structural field.

This report has been submitted to letter ballot of the committee which consists of 8 members; 8 members returned their ballots, of whom 6 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

M. O. WITHEY,
Chairman.

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[1] Compliance with this limitation would necessitate running the 200,000-lb. Riehle machine at The University of Wisconsin at free speeds of head not greater than 0.15 to 0.25 in. per min. for yield points between 10,000 and 40,000 lb. per sq. in. The appropriate free speeds to secure the requisite rates of strain or stress will vary with the type of machine, the condition of the grips, the area of cross-section of the specimen, and the load. Where the rate of stressing is used as a method of control it will be necessary to fix the rate of loading just before the limit of proportionality is passed.

DISCUSSION

Mr. Friedrich Körber[1] (*translation of discussion submitted by letter*).—We have read with considerable interest the report of the Research Committee on Yield Point of Structural Steel from which it develops that with the structural steels investigated the yield point and tensile strength had experienced no marked change as a result of aging following the hot rolling; that, however, the elongation, and in an especially marked degree, the reduction of area have been increased (8 and 30 per cent, respectively). No reasons are given in the report for this remarkable behavior of the steel; it is merely stated it would be an investigation worthy of study on the part of metallurgists to determine the reasons for this phenomenon.

Similar observations on rolled rail steel were made some years ago by F. Körber and J. Mehovar.[2] By means of longer aging or through short anneal at moderate temperatures, the elongation increased by about 30 per cent and the reduction of area by as much as 100 per cent, with practically no change in yield point and tensile strength. At that time the reason for the change in properties with aging and tempering was supposed to be due to relief of internal stresses. C. Drescher and R. Schäfer[3] could secure, by heating a steel casting for 60 hr. at a temperature of only 100 C., an increase in the elongation of from 7 to 24 per cent and in reduction of area of from 8 to 36 per cent. Their assumption that the lower values of the elongation and reduction of area could be attributed to the action of the hydrogen which had been driven off by means of the heating could be confirmed by tests on newly rolled rails tested at the Kaiser-Wilhelm-Institut für Eisenforschung.[4] As the accompanying Table I indicates, an obvious correlation exists between the improvement in ductility and the decrease of the hydrogen content of the material, from which it is deduced that hydrogen is the essential cause for the internal stresses, which were assumed in the earlier test results of the Eisenforschungsinstitut to be the cause of the decrease in ductility of newly rolled rails in comparison with rails which have been heated or aged for sufficiently long periods of time.

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[1] Director, Kaiser-Wilhelm-Institut für Eisenforschung, Düsseldorf, Germany.

[2] F. Körber and J. Mehovar, "Contribution to the Understanding of the Changes in Time and Mechanical Properties of Newly Rolled Rails, Particularly Those of Thomasstahl," *Mitteilungen*, Kaiser-Wilhelm-Institut für Eisenforschung, Vol. 17, p. 89 (1935).

[3] C. Drescher and R. Schäfer, Archiv für das Eisenhüttenwesen, Vol. 9, p. 327 (1935–1936).

[4] P. Bardenheuer, "Action of Hydrogen on Steel," *Stahl und Eisen*, Vol. 57, p. 593 (1937).

TABLE I.—DUCTILITY AND HYDROGEN CONTENT OF NEWLY ROLLED RAILS.

Steel	Test	Prior Treatment	Tensile Strength, lb. per sq. in.	Elongation (gage length = 10 diameters), per cent	Reduction of Area, per cent	Hydrogen, cu. cm. per 100 g.
Open-Hearth Steel..	No. 1	Tested immediately in as-received condition..........................	125 200	9.1	10	2.13
	No. 2	Specimen heated for 1 hr. at 200 C.....	126 200	13.1	20	0.10
	No. 3	Specimen aged for 7 weeks............	126 600	12.9	21	0.10
	No. 4	Pieces of rail aged for 7 weeks.........	122 300	12.3	19	0.37
Thomasstahl (Basic Bessemer Steel)..	No. 5	As-received condition (tested 5 days after rolling).......................	108 800	11.9	15	3.25
	No. 6	Specimen heated for 1 hr. at 200 C.....	109 100	18.1	34	0.48

MR. M. O. WITHEY[1] (*closure by letter*).—The Society is fortunate indeed to have the observations from the Kaiser-Wilhelm-Institut für Eisenforschung with regard to tests of steels under similar conditions to those mentioned in our aging experiments. These data from the Institut should be very valuable to any American metallurgists who may be interested in making similar studies.

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[1] Chairman, Research Committee on Yield Point of Structural Steel; Professor of Mechanics, University of Wisconsin, Madison, Wis.

REPORT OF COMMITTEE A-2

ON

WROUGHT IRON

Committee A-2 on Wrought Iron has held two meetings since the annual meeting of the Society in 1936, the first on October 21, 1936, in Philadelphia, Pa., and the second on March 3, 1937, in Chicago, Ill. A third meeting will be held prior to the presentation of this report to the Society when the report will be reviewed and other matters considered.

It is with regret that the committee records the death of two of its long-time members, J. C. Ramage, Engineer of Tests of the Southern Railway Co., and Thomas S. Wheelwright, President of the Old Dominion Iron and Steel Works; and of a comparatively new member, P. J. Neely, Chief Metallurgist, Railway Spring Division of the American Locomotive Co. Appropriate resolutions were sent to the Southern Railway Co. and to the Old Dominion Iron and Steel Works as well as to the families of Messrs. Ramage and Wheelwright. These resolutions appear elsewhere in this report.

Subcommittee II on Merchant Bar Iron and Subcommittee III on Staybolt and Engine-Bolt Iron will be consolidated. The work of these subcommittees has overlapped and it is believed that the work can be accomplished more efficiently and extensively by a single committee. The consolidated subcommittee will be designated Subcommittee II on Wrought-Iron Bars. Mr. J. Porter Gillespie of the Lockhart Iron and Steel Company, Pittsburgh, Pa., was elected chairman of this subcommittee.

Mr. E. L. Johnson, Engineer of Tests, New York Central Lines, 466 Lexington Avenue, New York City, was elected chairman of Subcommittee V on Chain Iron and Iron Chain succeeding Mr. F. M. Waring, resigned.

The resignation of Mr. E. S. Dixon, Metallurgist of the Texas Company, was accepted with regret at the March meeting.

Subsequent to the 1936 annual meeting, Committee A-2 presented to the Society on August 26, 1936, through Committee E-10 on Standards, a proposed revision of the Tentative Specifications for Wrought-Iron Plates (A 42 – 35 T), the modifications being merely for the purpose of clarifying the specifications. This recommendation was accepted[1] by Committee

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee A-2 reported results of the letter ballot vote as follows: Of a total membership of 57, 43 members returned their ballots, of whom 38 voted affirmatively, 0 negatively, and 5 marked their ballots "not voting."

E-10 and the specifications in their revised form appear in the 1936 *Proceedings*.[1]

During the year, two of the committee's specifications were submitted to the American Standards Association and approved as American standards, as follows:

Standard Specifications for Uncoated Wrought-Iron Sheets (A.S.T.M. A 162 - 36; A.S.A. No. G23-1937), and

Standard Specifications for Zinc-Coated (Galvanized) Wrought-Iron Sheets (A.S.T.M. A 163 - 36; A.S.A. No. G8.8-1937).

The Standard Specifications for Refined Iron Bars (A 41 - 36), approved originally in 1930 as American tentative standard, were submitted to the A.S.A. following their revision at the last annual meeting. The specifications in their revised form were approved as American tentative standard and bear the A.S.A. designation: G12-1936.

RECOMMENDATIONS AFFECTING STANDARDS

In this report the recommendations affecting standards and tentative standards are noted in brief form below together with the results of the letter ballot. The revisions are given in detail in the Appendix.[2]

In 1930, Committee A-2 adopted a definition for wrought iron[3] and deleted from the process clauses of the various specifications covering wrought-iron products, all reference to method of manufacture. At the October, 1936, meeting this former action was reversed and the process clauses are being revised, as indicated in the Appendix to this report, by the addition of the words, "pig puddled or processed wrought iron." Attention is called to the fact that no change is being made in the definition of wrought iron or in the note referring to iron scrap, both of which are referred to in the process clauses.

The purpose of the revisions being proposed in the sections providing for the number of tests under the Tentative Specifications for Single and Double Refined Wrought-Iron Bars (A 189 - 36 T) and in the Standard Specifications for Staybolt Wrought Iron, Solid (A 84 - 36) and for Staybolt Wrought Iron, Hollow Rolled (A 86 - 36) is to eliminate what the committee believes to be an unnecessary number of tests. These sections at present stipulate that bars shall be grouped into lots of 100 each according to bar size and that the physical tests specified be made on each lot. Where only a few bars of several different sizes are offered for inspection at the same time, the testing of each size individually is considered to be unnecessary from the standpoint of a determination of the quality of the material and is a burden on the producer.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 652 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 204.

[2] See pp. 107, 108 and 1307 to 1310.

[3] 1936 Book of A.S.T.M. Standards, Part I, p. 453.

I. Proposed Revisions of Tentative Standards.—Revisions as given in the Appendix are recommended in the following tentative specifications, the revised specifications to be continued as tentative:

Tentative Specifications for:
Wrought-Iron Plates (A 42 – 36 T),
Wrought-Iron Rivets and Rivet Rounds (A 152 – 36 T), and
Single and Double Refined Wrought-Iron Bars (A 189 – 36 T).

II. Proposed Revisions of Standards.—The committee recommends that the revisions as appended hereto[1] in the following specifications be accepted for publication as tentative:

Standard Specifications for:
Iron and Steel Chain (A 56 – 30),
Welded Wrought-Iron Pipe (A 72 – 33),
Wrought-Iron Rolled or Forged Blooms and Forgings (A 73 – 36),
Staybolt Wrought Iron, Solid (A 84 – 36),
Staybolt Wrought Iron, Hollow Rolled (A 86 – 36),
Uncoated Wrought-Iron Sheets (A 162 – 36), and
Zinc-Coated (Galvanized) Wrought-Iron Sheets (A 163 – 36).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 57 members; 41 members returned their ballots, the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. PROPOSED REVISIONS OF TENTATIVE STANDARDS			
Specifications for Wrought-Iron Plates (A 42 – 36 T)	36	1	4
Specifications for Wrought-Iron Rivets and Rivet Rounds (A 152 – 36 T)	32	0	9
Specifications for Single and Double Refined Wrought-Iron Bars (A 189 – 36 T)	36	0	5
II. PROPOSED REVISIONS OF STANDARDS			
Specifications for Iron and Steel Chain (A 56 – 30)	38	1	2
Specifications for Welded Wrought-Iron Pipe (A 72 – 33)	37	1	3
Specifications for Wrought-Iron Rolled or Forged Blooms and Forgings (A 73 – 36)	33	1	7
Specifications for Staybolt Wrought Iron, Solid (A 84 – 36)	29	0	12
Specifications for Staybolt Wrought Iron, Hollow Rolled (A 86 – 36)	35	1	5
Specifications for Uncoated Wrought-Iron Sheets (A 162 – 36)	29	0	12
Specifications for Zinc-Coated (Galvanized) Wrought-Iron Sheets (A 163 – 36)	29	0	12

RESOLUTIONS ON DEATHS OF MEMBERS

The resolutions adopted by Committee A-2 on the deaths of two of its members are given below:

J. C. RAMAGE

WHEREAS, through death on October 21, 1936, we have lost our friend and co-worker, J. C. Ramage, a member of this committee since its formation in 1909, and

WHEREAS, his experience in the testing of materials, his wisdom, judgment and farsightedness were invaluable to his fellow members, particularly during the early years of committee work when character and talents such as he possessed were most needed, and

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[1] See pp. 1307 to 1310.

WHEREAS, his unfailing courtesy and kindliness endeared him to his fellow members, therefore,

BE IT RESOLVED, that the members of Committee A-2 on Wrought Iron of the American Society for Testing Materials, in meeting assembled this third day of March, 1937, express their deep regret at his passing and extend sincere sympathy to those bereaved, and

BE IT FURTHER RESOLVED, that this resolution be spread upon the minutes of this meeting and that copies be sent to the family of Mr. Ramage and to the Southern Railway Co. of which he was Engineer of Tests.

THOMAS S. WHEELWRIGHT

WHEREAS, through death on December 31, 1936, we have lost our friend and co-worker, Thomas S. Wheelwright, a member of this committee since 1925, and

WHEREAS, his kindly humor, his genuine interest in the affairs of the committee and his unfailing courtesy will long be remembered by his fellow members, therefore,

BE IT RESOLVED, that the members of Committee A-2 on Wrought Iron of the American Society for Testing Materials, in meeting assembled this third day of March, 1937, express their deep regret at his passing and extend sincere sympathy to those bereaved, and

BE IT FURTHER RESOLVED, that this resolution be spread upon the minutes of this meeting and that copies be sent to the family of Mr. Wheelwright and to the Old Dominion Iron and Steel Works of which he was President.

This report has been submitted to letter ballot of the committee which consists of 57 members; 41 members returned their ballots, of whom 40 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

J. B. YOUNG,
Chairman.

G. H. WOODROFFE,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee A-2 on Wrought Iron presented to the Society on August 26, 1937, through Committee E-10 on Standards the following recommendations:

Proposed Tentative Specifications for Rolled Wrought-Iron Shapes and Bars, and
Proposed Tentative Revisions of Standard Specifications for:
  Iron and Steel Chain (A 56 – 30),
  Staybolt Wrought Iron, Solid (A 84 – 36), and
  Staybolt Wrought Iron, Hollow Rolled (A 86 – 36).

The above recommendations were accepted by Committee E-10 and appear on pp. 649, 1307, 1308 and 1310, respectively.

APPENDIX

PROPOSED REVISIONS IN TENTATIVE STANDARDS FOR WROUGHT IRON

In this Appendix are given proposed revisions of specifications covering wrought iron and its products, as referred to earlier in this report. In connection with each title is given the reference to the publication in which the specifications appear in their present form.

Proposed Revisions of Tentative Standards

Proposed Revision of Tentative Specifications for Wrought-Iron Plates (A.S.T.M. Designation: A 42 – 36 T):[1]

Section 2.—Change from its present form: namely,

> 2. (*a*) All plates shall be rolled from blooms, piles or slabs which are entirely free from any admixture of iron scrap (Note) or steel.
>
> (*b*) Piles for plates shall be made from wrought iron. The constituent bars shall be the full length of the pile.

to read as follows:

> 2. Plates shall be rolled from blooms, piles or slabs made from all pig puddled or processed wrought iron, free from any admixture of iron scrap (Note) or steel. The component parts of piles shall be of the full length of the pile.

Proposed Revision of Tentative Specifications for Wrought-Iron Rivets and Rivet Rounds (A.S.T.M. Designation: A 152 – 36 T):[1]

Section 1.—Change to read as follows by the addition of the italicized words and the omission of the word in brackets:

> 1. The rivet bars shall be made from *all pig puddled or processed* wrought iron, [which] *and* shall be free from any admixture of iron scrap or steel.

Proposed Revision of Tentative Specifications for Single and Double Refined Wrought-Iron Bars (A.S.T.M. Designation: A 189 – 36 T):[1]

Section 2.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> 2. (*a*) The bars shall be made from pig puddled or processed wrought iron and shall be free from any admixture of iron scrap or steel. *In the final piling* all bars shall be of the full length of the pile.
>
> Grade A.—The original muck bars shall be [twice] piled, *rolled, repiled* and rerolled.
>
> Grade B.—The original muck bars shall be piled and [rerolled] *rolled.*

Section 5.—In the table of physical properties change footnote "a," which refers to the elongation requirement of Grade A bars under $1\frac{5}{8}$ in. in diameter, to read as follows by the addition of the italicized words and figures and the omission of the figure in brackets. Also, add the reference letter "a" after the elongation requirement of Grade B bars under $1\frac{5}{8}$ in. in diameter:

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 652, 658, 648 (1936); also 1936 Book of A.S.T.M. Tentative Standards, pp. 204, 210, 200.

> [a] Bars not exceeding [$\frac{5}{8}$] *7/8* in. in diameter or thickness will be accepted if the elongation in 8 in. is not less than 25 per cent *for Grade A and 22 per cent for Grade B material.*

Add a footnote "b" to read as follows, the reference letter to be added after the lines "tensile strength, lb. per sq. in." for Grades A and B bars:

> [b] A deduction of 1000 lb. per sq. in. shall be allowed from the minimum tensile strength specified for specimens reduced by machining.

Section 9.—Change the last sentence in Paragraph (*a*) to read as follows by the addition of the italicized words and the omission of the words and figure in brackets:

> For bars over $2\frac{1}{2}$ in. in diameter or thickness, the axis of the tension specimen shall be located at any point one-half the distance from the center to the surface and shall be parallel to the axis of the bar; and the specimen [shall] *may* be turned to [the] *a convenient* diameter [of 1 in.] for a length of at least 9 in. with enlarged ends.

Change the third sentence in Paragraph (*b*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> For flat bars the bend [specimen] *specimens* may be reduced to a width not less than [their] *the* thickness *of the bar.*

Add a new sentence between the present third and fourth sentences to read as follows:

> In any case, however, it is permissible to reduce the bend test specimen for flat bars to at least a 1-in. square.

Section 10.—Add a new Paragraph (*b*) to read as follows, relettering the present Paragraph (*b*) as Paragraph (*c*):

> (*b*) Where small lots of different sizes are to be tested the bars shall be grouped into lots of 5000 lb., or fraction thereof, without regard to size, and one bar representing each lot of 5000 lb., or fraction thereof, shall be tested as specified in Sections 4, 5, 6, 7 and 8.

REPORT OF COMMITTEE A-3

ON

CAST IRON

Since the last annual meeting of the Society, Committee A-3 on Cast Iron has held two meetings, one in Cleveland, Ohio, on October 20, 1936, and the other in Detroit, Mich., on March 3, 1937. A third meeting will be held during the coming annual meeting of the Society.

It is with regret that the committee records the loss through death of the following members: Dr. F. C. Langenberg, Vice-President of the United States Pipe and Foundry Co., who served as chairman of Subcommittee I on Pig Iron; Mr. N. S. Hill, Jr., consulting engineer; and Mr. J. C. Ramage, Engineer of Tests of the Southern Railway Co.

One member of the committee has resigned and two have been elected to membership, which brings the total personnel of the committee to 101 members, of whom 27 are classified as producers, 42 as consumers, and 32 as general interest members.

Subsequent to the 1936 annual meeting, Committee A-3 presented to the Society through Committee E-10 on Standards, proposed Tentative Definitions of Terms Relating to Cast Iron (A 196 – 36 T) which had been prepared by Subcommittee X on Nomenclature and Definitions (Robert Job, chairman). The proposed tentative definitions were accepted[1] by Committee E-10 on August 26, 1936, and appear in the 1936 *Proceedings*.[2]

It is recommended that the present tentative standards under the jurisdiction of Committee A-3 be continued as tentative without revision.

Subcommittee IV on Car Wheels (F. K. Vial, chairman) is at work on a complete revision of the Tentative Specifications for Chilled-Tread Cast-Iron Wheels (A 46 – 30 T). These specifications are to be rearranged and when completed the revised specifications will be in close agreement with the specifications for car wheels developed by the Association of American Railroads.

Subcommittee VI on General Castings (J. W. Bolton, chairman) has under consideration the establishment of deflection values in the flexure test for inclusion in the Standard Specifications for Gray-Iron Castings (A 48 – 36).

Subcommittee XI on Methods of Testing (R. S. MacPherran, chairman) has been granted an appropriation of $200 by the Society's Committee E-9 on Research for research work on fatigue tests of cast iron being carried out by J. B. Kommers of the University of Wisconsin. The subcommittee will collaborate with Professor Kommers in this work.

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[1] In submitting these tentative definitions to Committee E-10 on Standards, Committee A-3 reported results of the letter ballot vote as follows: Out of a total membership of 102, 70 members returned their ballots, of whom 67 voted affirmatively, 1 negatively, and 2 members marked their ballots "not voting."

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 667 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 236.

Subcommittee XV on Impact Testing (J. T. MacKenzie, chairman) has done sufficient work on impact testing to warrant making this information available to other members who are interested in conducting tests along the same lines. However, before doing so, a standard drop test machine must be adopted. The subcommittee plans to contact manufacturers of testing equipment for information regarding the quality of mechanical devices for this machine.

This report has been submitted to letter ballot of the committee which consists of 101 members; 63 members returned their ballots, of whom 61 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

W. H. ROTHER,
Chairman.

H. C. AUFDERHAAR,
Secretary.

REPORT OF COMMITTEE A-5

ON

CORROSION OF IRON AND STEEL

Committee A-5 on Corrosion of Iron and Steel has held two meetings during the past year; one on June 30, 1936, during the annual meeting of the Society at Atlantic City, N. J., and the other on March 3, 1937, at the spring group meetings of A.S.T.M. committees in Chicago, Ill.

Since the last report, 5 new members have been elected and 1 member has died; the committee membership now totals 126, of whom 57 are classified as producers, 46 as consumers, and 23 as general interest members.

RECOMMENDATIONS AFFECTING STANDARDS

Proposed Revisions of Standards.—The committee recommends that the revisions proposed in the following two standard specifications be accepted for publication as tentative in the form of two new tentative specifications, as appended hereto:[1]

Standard Specifications for:

Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (A 116 – 30),[2] and
Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (A 121 – 30).[3]

The requirements of the proposed revised specifications differ from those prescribed in the present standard specifications primarily in the inclusion of three weight-of-coating classes for both fencing and barbed wire.

These recommendations have been submitted to letter ballot of the committee which consists of 126 members; 124 members returned their ballots, the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
PROPOSED TENTATIVE REVISIONS OF STANDARDS			
Specifications for Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (A 116 – 30)	82	0	42
Specifications for Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (A 121 – 30)	82	0	42

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[1] See pp. 646 and 643.
[2] 1936 Book of A.S.T.M. Standards, Part I, p. 368.
[3] *Ibid.*, p. 362.

TABLE I.—RECORD OF FAILURES IN ATMOSPHERIC EXPOSURE TESTS AT ANNAPOLIS OF 22-GAGE COPPER-BEARING AND NON-COPPER-BEARING CORRUGATED BLACK SHEETS.

Sheets Exposed October 17, 1916. Inspections Made Semiannually from 1917 to 1937.

Group	Designation	Series	Average Analysis, per cent						Number of Sheets in Test	Life of Each Failed Sheet in Years																Number of Sheets in Test	Number of Sheets Failed	Average Life of Sheets in Years	Group
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper																					
Z.....	Open-hearth steel	B	0.032	0.32	0.063	0.042	.....	0.019	3	..	..	..	..	..	..	..	..	..	..	..	..	..	13.5	17.0	19.0	3	All	16.5	.....Z
O.....	Open-hearth steel	A	0.121	0.536	0.008	0.030	0.249	0.020	12	..	..	..	..	7.0	9.0	9.0	9.0	9.0	9.5	10.0	10.0	11.0	11.0	13.0	17.0	12	All	10.4	.....O
B.....	Open-hearth steel	A	0.09	0.38	0.017	0.062	0.036	0.084	3	..	..	..	..	..	..	..	..	..	..	..	..	..	20.0	*	*	3	1	....	.....B
M....	Open-hearth steel	B	0.093	0.419	0.013	0.041	0.011	0.185	14	..	..	20.0	*	*	*	*	*	*	*	*	*	*	*	*	*	14	1	....	.....M
B.....	Open-hearth steel	A	0.06	0.27	0.014	0.053	0.006	0.187	9	..	..	..	..	..	..	..	19.5	20.0	*	*	*	*	*	*	*	9	2	....	.....B
Z.....	Open-hearth steel	B	0.067	0.351	0.045	0.052	0.003	0.187	3	..	..	..	..	..	..	..	..	..	..	..	..	..	*	*	*	3	None	....	.....Z
T.....	Open-hearth steel	B	0.083	0.313	0.009	0.020	0.005	0.227	14	..	..	19.5	19.5	19.5	*	*	*	*	*	*	*	*	*	*	*	14	3	....	.....T
K.....	Open-hearth steel (acid)	A	0.107	0.447	0.091	0.046	0.004	0.237	16	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	16	None	....	.....K
H.....	Open-hearth steel	A	0.069	0.387	0.016	0.027	0.004	0.244	16	19.5	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	16	1	....	.....H
Z.....	Open-hearth steel	B	0.065	0.35	0.050	0.059	0.003	0.264	3	..	..	..	..	..	..	..	..	..	..	..	..	..	*	*	*	3	None	....	.....Z
Z.....	Open-hearth steel	B	0.073	0.42	0.052	0.054	Trace	0.641	3	..	..	..	..	..	..	..	..	..	..	..	..	..	*	*	*	3	None	....	.....Z
S.....	Open-hearth iron	B	0.02	0.023	0.006	0.022	0.004	0.022	14	..	..	7.0	8.0	8.5	9.0	9.5	10.0	11.0	11.5	11.5	12.5	12.5	12.5	12.5	12.5	14	All	10.6	.....S
D....	Open-hearth iron	A	0.02	0.026	0.004	0.022	0.002	0.024	7	..	..	..	..	..	..	..	..	..	16.0	16.0	16.0	19.5	19.5	*	*	7	5	....	.....D
X....	Open-hearth iron	B	0.021	0.055	0.007	0.031	0.004	0.027	14	..	..	10.5	13.0	13.5	14.5	14.5	16.0	16.0	16.5	16.5	17.0	17.5	17.5	18.0	19.0	14	All	15.7	X
C.....	Open-hearth iron	A	0.01	0.028	0.002	0.027	0.002	0.141	3	..	..	..	..	..	..	..	..	..	..	..	..	..	17.5	*	*	3	1	....	C
C.....	Open-hearth iron	A	0.015	0.028	0.006	0.036	0.003	0.194	9	..	..	..	..	..	..	..	16.5	16.5	18.5	19.0	19.0	20.0	*	*	*	9	6	....	C
U.....	Open-hearth iron	B	0.017	0.074	0.008	0.037	0.005	0.260	14	..	..	15.5	16.5	19.0	19.0	19.0	19.0	19.0	19.5	20.0	20.0	*	*	*	*	14	10	....	.....U
V.....	Open-hearth iron	B	0.018	0.051	0.007	0.021	0.005	0.304	14	..	..	17.0	17.0	17.5	18.5	18.5	19.0	20.0	20.0	*	*	*	*	*	*	14	8	....	V
A.....	Bessemer steel	A	0.038	0.386	0.089	0.040	0.007	0.014	15	..	15.5	16.5	16.5	17.0	17.0	17.0	18.0	19.0	19.0	19.5	19.5	19.5	19.5	19.5	20.0	15	All	18.2	.....A
Z.....	Bessemer steel	B	0.063	0.41	0.102	0.044	0.009	0.013	3	..	..	..	..	..	..	..	..	..	..	..	..	..	11.5	11.5	11.5	3	All	11.5	.....Z
Z.....	Bessemer steel	B	0.077	0.44	0.095	0.036	0.003	0.140	3	..	..	..	..	..	..	..	..	..	..	..	..	..	*	*	*	3	None	....	.....Z
I.....	Bessemer steel	A	0.041	0.365	0.097	0.068	0.008	0.252	16	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	16	None	....	I
Z.....	Bessemer steel	B	0.058	0.37	0.093	0.043	0.009	0.269	3	..	..	..	..	..	..	..	..	..	..	..	..	..	*	*	*	3	None	....	.....Z
Z.....	Bessemer steel	B	0.055	0.38	0.110	0.046	.....	0.527	3	..	..	..	..	..	..	..	..	..	..	..	..	..	*	*	*	3	None	....	.....Z
Y.....	Wrought iron	B	0.03	0.055	0.139	0.021	0.218	0.020	3	..	..	..	..	..	..	..	..	..	..	..	..	..	18.0	19.5	*	3	2	....	.....Y
E.....	Wrought iron	A	0.033	0.034	0.114	0.021	0.134	0.283	11	..	..	..	..	..	19.5	*	*	*	*	*	*	*	*	*	*	11	1	....	.....E
							Total..........		228																Total....	228	102		

* Indicates unfailed sheets.

Activities of Subcommittees

Subcommittee III on Inspection of Annapolis Tests (E. S. Taylerson, chairman).—Since the inspection on November 4, 1935, the subcommittee has held two meetings at Annapolis, Md. On April 27, 1936, 15 failures were observed, namely, A6, A8, A9, A13, A14 (non-copper bessemer steel), B4 (copper-bearing open-hearth steel), D5, D12 (non-copper pure iron), E8 (copper-bearing puddled iron), B4 (copper-bearing basic open-hearth steel), T1, T3, T10 (copper-bearing open-hearth steel), U11 (copper-bearing pure open-hearth iron), and Y1 (non-copper wrought iron). On October 16, 1936, 9 failures were observed, namely, A12 (non-copper bessemer steel), B7 (copper-bearing open-hearth steel), B27 (non-copper open-hearth steel), C23 (copper-bearing pure open-hearth iron), M14 (copper-bearing open-hearth steel), U4, U8 (copper-bearing pure open-hearth iron), and V11, V12 (copper-bearing pure open-hearth iron). All of the failures recorded up to this time are in the No. 22 gage material. A summary of the failures to date is given in the accompanying Table I.

At the inspection held in October, 1936, the committee celebrated the twentieth anniversary of these atmospheric corrosion tests at a banquet held at Carvel Hall, Annapolis. Some of the men who constituted the original A.S.T.M. committee in charge of this long-time test program and a number of others now active in the inspection and several invited guests were present.

Subcommittee V on Total Immersion Tests (W. J. Jeffries, chairman).—Although no meetings of the subcommittee have been held during the past year, the routine inspections of the copper-bearing and non-copper-bearing steel sheet specimens submerged in sea water at the U. S. Navy Yards at Key West, Fla., and Portsmouth, N. H., have been made during the year. At Key West there are still 18 specimens remaining on the test racks, 14 having failed since the 1936 report.[1] The 1936 report[1] of this subcommittee showed 2 specimens remaining at Portsmouth. At the inspection on September 8, 1936, the last specimen exposed at Portsmouth was found to have failed, and the exposure test at this location has, therefore, been completed. The failures observed at the inspections made during the past year are given below:[1]

Failures at Key West

(No. 16 Gage Sheets Exposed March 1, 1927, in Sea Water)

Specimen	Rack	Dates of Inspection
CC–12	6	April 24, 1936
MM–9	3	April 24, 1936
CC–8	3	June 25, 1936
EE–9	4	September 25, 1936
CCC–17	5	October 26, 1936

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[1] For detailed tabulations of failures observed in the total immersion tests at Key West, Fla., and Portsmouth, N. H., see Tables I and II (Plates I and II) of the 1936 Report of Committee A-5, *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 104 (1936).

FAILURES AT KEY WEST (*Continued*)

(No. 16 Gage Sheets Exposed March 1, 1927, in Sea Water)

SPECIMEN	RACK	DATES OF INSPECTION
UU-4	3	January 22, 1937
DD-12	2	January 22, 1937
XX-7	2	February 24, 1937
VV-35	6	March 24, 1937
FF-11	4	March 24, 1937
CCC-12	2	March 24, 1937
TT-1	1	March 24, 1937
DD-21	4	April 26, 1937
CC-7	2	June 25, 1937

FAILURES AT PORTSMOUTH

(No. 16 Gage Sheets Exposed March 7, 1927, in Sea Water)

CCC-34	6	August 5, 1936
VV-51	5	September 8, 1936

The steel pipe specimens immersed at both of the above locations appeared to be in the same condition as was described in the report of the committee last year, at which time general corrosion and pitting were reported to be uniform over all the specimens. The tests have not progressed to a point where conclusions may be drawn.

The riveted corrosion test plates submerged at Portsmouth and Key West have undergone the usual inspection. Copper replicas will be made as usual.

Subcommittee VI on Specifications for Metallic-Coated Products (W. H. Finkeldey, chairman).—Subcommittee VI has held two meetings during the past year. It has prepared and presented to Committee A-5 proposed tentative revisions of the Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (A 116 – 30) and for Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (A 121 – 30) in the form of two new tentative specifications, appended hereto.[1]

The Section on Strand is preparing proposed revisions of the Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Wire Strand (Cable) (A 122 – 33). It is proposed to reduce the number of sizes of strand shown in Table II of the present specifications, to increase the weight of zinc coatings shown in Table I, to revise the minimum number of dips in the Preece test, to revise the tables of physical properties of strand, and to revise the text to conform to these changes.

The Section on Chain Link Fence is working on proposed revisions of the Standard Specifications for Zinc-Coated Iron or Steel Chain-Link Fence Fabric Galvanized After Weaving (A 117 – 33) and on the preparation of proposed specifications for chain-link fence fabric galvanized before weaving. It is expected that the new and revised specifications will be submitted to Subcommittee VI in June.

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[1] See pp. 646 and 643.

The Section on Plated Coatings is awaiting the development of methods of determining the thickness of coatings before proposing any revisions in the Tentative Specifications for Electrodeposited Coatings of Zinc on Steel (A 164 – 35 T), Cadmium on Steel (A 165 – 35 T), and Nickel and Chromium on Steel (A 166 – 35 T).

The Section on Line Wire plans to review the Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Telephone and Telegraph Line Wire (A 111 – 33) and to prepare such revisions as are desirable to bring these specifications in line with improvements in the manufacture of these materials.

There has been considerable discussion of the aluminum limit of 0.01 per cent in the bath prescribed in the Standard Specifications for Zinc (Hot-Galvanized) Coatings on Structural Steel Shapes, Plates and Bars and Their Products (A 123 – 33). Apparent difficulties in meeting this requirement have been traced to inaccurate analytical methods. Both chemical and spectrographic methods of analysis of sufficient accuracy are available, and since no reference is made in the specifications to a proper analytical method, it is proposed to revise the specifications to include such a reference.

The Tentative Specifications for Zinc Coating (Hot-Dip) on Hardware and Fastenings (A 153 – 33 T) are to be continued as tentative for another year. It is proposed to revise these specifications to include reference to a test method for determining the weight or thickness of coating when the development of this subject has been completed by Subcommittee VII.

Subcommittee VII on Methods of Testing (L. W. Hopkins, chairman).—Subcommittee VII has held one meeting during the year, at Atlantic City in June, 1936. Other meetings have been held by the sections working on specific projects.

The subcommittee cooperated in the preparation and presentation of the Symposium on Corrosion Testing Procedure,[1] held on March 2, 1937, in Chicago, as this is the type of work with which Subcommittee VII is directly concerned.

Comments have been received on the Tentative Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Wire (A 191 – 36 T) and are being studied by the section working on the Preece test.

Methods for determining the weight of coating on zinc-coated hardware have been studied by the Section on Hardware and it is expected that these will be ready for presentation in June to the subcommittee for submission as a tentative standard.

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[1] Symposium on Corrosion Testing Procedure, Chicago Regional Meeting, Am. Soc. Testing Mats. (1937). (Available as separate publication.)

The Section on Test Methods for Determining the Weight or Thickness of Plated Coatings has studied several methods, including metallographic, stripping, the chord method, dropping, jet, and spot tests, and a newly developed magnetic method for nickel coatings. Before attempting to prepare standards it is planned to compile and distribute these methods in the hope that further comparative work will be done and comments received.

Subcommittee VIII on Field Tests of Metallic Coatings (R. F. Passano, chairman).—Subcommittee VIII has held one meeting since the last annual meeting of the Society. Inspection of the galvanized sheets and coated hardware exposed at five test locations is being continued periodically. Since the developments in these two field tests are not now so rapid as they were, it did not appear advisable to devote this year's report to them. Extensive data and information concerning these two investigative studies have been made available in earlier reports;[1] at present there is no great change in either the amount of data or the indications of the exposure tests.

The principal progress made by Subcommittee VIII during the year has been in connection with the third and last of the authorized field tests on its original program, namely, the field tests on wire and wire products. A detailed account of the erection of the specimens involved in these tests is given in the Report of the Wire Test Committee which appears as an Appendix to this report.

Subcommittee X on Embrittlement Investigation (W. G. Kelley, chairman).—Subcommittee X has practically completed its work and has held no meetings during the year.

This report has been submitted to letter ballot of the committee which consists of 126 members; 124 members returned their ballots, of whom 116 have voted affirmatively and 8 negatively.

Respectfully submitted on behalf of the committee,

F. F. FARNSWORTH,
Chairman.

T. R. GALLOWAY,
Secretary.

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[1] For the two latest detailed reports on these investigations, see *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 107 (1936), and Vol. 35, Part I, p. 88 (1935).

REPORT OF THE WIRE TEST COMMITTEE

The complete early history of the wire test program, its scope and methods of procedure and the personnel responsible for its carrying out were published in the 1934 report[1] of Subcommittee VIII on Field Tests of Metallic Coatings. In the Appendix to the 1934 report was given a detailed breakdown of the types of materials to be tested and the numbers of samples to be exposed. The program, as outlined in the 1934 report, has been followed very closely and the committee is pleased to report the virtual completion of the phase of the wire test program for which it has been responsible.

Attached to this 1937 report is an Appendix, drawn up along the lines of the Appendix previously given in the 1934 report, showing the exact numbers and types of specimens which have been exposed, their distribution to the various test sites and their location in the test plot. The Appendix as now presented carries all of the data available on the test materials exposed which have been obtained to date. Complete chemical and physical characterizations of the original materials provided for by this committee in the referee testing which is to be done by the National Bureau of Standards will not be available for several months but will be published as a part of the Subcommittee VIII report at the earliest possible moment.

The major portion of the work required subsequent to the organization of the testing program by the Wire Test Committee has been done by Mr. C. L. Warwick, Secretary-Treasurer of the Society, who has acted as Director of Tests, and by Mr. H. E. Smith, who has functioned as Engineer of Tests. It will be realized that the magnitude of the work made it impossible to carry on under normal committee routines and that the full time of a capable engineer was required to adequately supervise the job. The committee wishes here to formally acknowledge the extremely efficient and capable manner in which the Director and Engineer of Tests have carried out the complicated task handed to them and to compliment them on the type of job they have performed and the precision with which it has been carried out.

The committee wishes to state its appreciation of the importance of the following factors in making possible a test program of this type:

> It has been able to function under the direction of the American Society for Testing Materials which has made available its resources and organization for the prosecution of this work.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 159 (1934).

It has enjoyed the enthusiastic and generous cooperation of the various state agricultural colleges which have set aside for a period of 20 yr. a test site for this work, have appointed a staff member to be in responsible charge of the test site and to represent the American Society of Agricultural Engineers on inspections of tests to be made at each particular location, and have furnished the protective enclosures for the test site, and erected the test racks within the enclosure. These colleges have, in addition, agreed to maintain the test site over the expected test period of 20 yr. This type of cooperation has relieved the committee of a tremendous amount of responsibility and expense and has made available to it test facilities widely scattered throughout the United States which could have been obtained in no other way.

Interested producers of the materials under test have been generously willing to prepare special heats of material as required by the committee, to carefully test these products in their own laboratories and furnish these data to the committee, and to assign responsible engineers to the job of aiding in the planning and prosecution of the test program and in the long and involved inspection program which must follow the inauguration of the tests.

Vitally necessary financial assistance has been realized through the generous contribution of funds required to defray the necessary expenses of the tests made by interested consumer organizations. Typical expenses which have required such contributions are: warehouse rental, salaries of the Engineer of Tests and his helpers, trucking, freight, the purchase of incidental materials for the test racks, and salaries of the engineers carrying out the referee tests at the Bureau of Standards.

The committee at this time wishes to formally acknowledge the assistance given to it by the various cooperative agencies listed below:

Mr. C. L. Warwick, Director of Tests
  Secretary-Treasurer of the American Society for Testing Materials
Mr. H. E. Smith, Engineer of Tests
Professor H. W. Riley, American Society of Agricultural Engineers, Official representative on Committee A-5
The Cooperating Universities, Colleges, and Experiment Stations:
  Cornell University, Ithaca, N. Y.
    Professor H. W. Riley
  Iowa State College, Ames, Iowa
    Professor J. B. Davidson
  Kansas State College, Manhattan, Kans.
    Professor F. C. Fenton
  Pennsylvania State College, State College, Pa.
    Professor R. U. Blasingame
  Purdue University, Lafayette, Ind.
    Professor William Aitkenhead

Texas Agricultural Experiment Station, College Station, Tex.
  Professor H. P. Smith
University of California, Agricultural College, Davis, Calif.
  Professor J. D. Long

Manufacturers who have furnished test materials without charge to the committee, as follows:

American Steel and Wire Co.
Anchor Post Fence Co.
Bethlehem Steel Co., Inc.
Continental Steel Corp.
Copperweld Steel Co.
Cyclone Fence Co.
Indiana Steel and Wire Co.
Keystone Steel and Wire Co.
Page Steel and Wire Division of American Chain and Cable Co., Inc.
Pittsburgh Steel Co.
John A. Roebling's Sons Co.
Wheeling Metal and Manufacturing Co.
Wickwire Spencer Steel Co.
Williamsport Wire Rope Co.

Manufacturers who have furnished, without charge or at cost, structural materials for supports, fabricating service or erection service, as follows:

American Sheet and Tin Plate Co.
American Steel and Wire Co.
Anchor Post Fence Co.
Bayonne Bolt Works
Bethlehem Steel Co., Inc.
Carnegie-Illinois Steel Corp.
Continental Steel Corp.
Copperweld Steel Co.
Cyclone Fence Co.
Diamond Expansion Bolt Co., Inc.
Frick-Reid Supply Corp.
Hanlon-Gregory Galvanizing Co.
Hubbard and Co.
Inland Steel Co.
Keystone Steel and Wire Co.
John W. Masury and Son
Page Steel and Wire Co.
Pittsburgh Steel Co.
Republic Steel Corp.
Western Union Telegraph Co.
Wickwire Spencer Steel Co.
Youngstown Sheet and Tube Co.

The committee is also indebted to the Duquesne Light Co., Pittsburgh, Pa., and the Quartermaster's Office, Fort Hancock (Sandy Hook), N. J., for services made available in connection with the erection of samples at those two locations; also to the City of Santa Cruz, Calif., for making available to the committee a very suitable test site for the desired period of 20 yr.

Organizations which have contributed funds for the support of this project. The original contributors to the A-5 research fund designed to carry out outdoor exposure tests on galvanized sheets, hardware and wire were as follows: (This list was reported previously in the April 30, 1925, *Bulletin* of the Society.)

American Chain Co.
American Malleable Castings Association
American Metal Co., Ltd.
American Rolling Mill Co.
American Sheet and Tin Plate Co.
American Steel and Wire Co.
American Zinc Institute, Inc.
Babcock and Wilcox Co.
Bethlehem Steel Co., Inc.
Birdsboro Steel and Foundry Co.
A. M. Byers Co.
H. Y. Carson
Central Steel Co.

Crane Co.
Eagle-Picher Lead Co.
Electro-Sherardizing Co.
Finn Metal Works, Jno.
General Electric Co.
Hanson and Van Winkle Co.
International Derrick and Equipment Co.
International Harvester Co.
Keystone Steel and Wire Co.
Koppers Co.
Lukens Steel Co.
National Tube Co.
New Jersey Zinc Co.
Newport Rolling Mill Co.
Ohio Brass Co.
Pittsburgh Electro Galvanizing Co.
Reading Iron Co.
Republic Iron and Steel Co.
Robinson and Co., Dwight P.
Roebling's Sons Co., John A.
St. Joseph Lead Co.
Sheet Steel Trade and Extension Committee
Standard Oil Co. (New Jersey)
Stanley Works
Tennessee Coal, Iron and Railroad Co.
The Texas Company
Tidewater Oil Co.
United Alloy Steel Corp.
U. S. Chain and Forging Co.
Vacuum Oil Co.
Western Electric Co., Inc.
Western Union Telegraph Co.
Westinghouse Electric and Manufacturing Co.
Wheeling Steel Corp.
Wood Iron and Steel Co., Alan
York Manufacturing Co.
Youngstown Sheet and Tube Co.

Recent contributions made to enable the completion of the wire test program (greatly enlarged over its original scope) have been made by the following organizations:

The American Zinc Institute	$2000
Utilities Coordinated Research, Inc	6000

The committee is particularly indebted to these two organizations for their extremely generous financial assistance. As a result of the availability of these funds, it has been possible for the committee to arrange with the National Bureau of Standards to have all referee testing of the original chemical and physical properties of the test materials done at one laboratory by one group of test engineers. The committee feels that in no other way could it obtain the precise data on original samples necessary for the drawing of adequate technical conclusions at the completion of the test.

The accounting of committee funds for this test program is as follows: When the wire test program was begun, the committee had available for its entire corrosion test program $8678.57, which included the $2000 contribution from the American Zinc Institute but not the later contribution of the Utilities Coordinated Research, Inc. Expenditures for the wire tests, up to and including February 4, 1937, totaled $7159.51. The major expense items involved the salary of the Engineer of Tests, purchase of support material, wages for labor and similar charges in connection with fabrication and erection of samples, and shipping costs on support and test materials. These expenditures, together with $1500 ear-marked by Committee A-5 for the maintenance of other test projects, left available on February 4, 1937, for the completion of the wire test project a total of $19.06. Since that time, the Utilities Coordinated Research, Inc., have contributed an additional $6000 to carry these tests to completion. Of this $6000, $4000 will be spent during the coming year at the National Bureau of Standards to pay the salaries of two research associates engaged to carry out the referee testing program designed to determine the original chemical and physical properties of the test materials. The remainder of the fund, although small, will be required to defray necessary expenses involved in the inspection of samples over a period of years.

The committee is glad to report, through the efforts of Dr. L. J. Briggs Director, and Dr. H. S. Rawdon, Metallurgist, of the National Bureau of Standards, that Mr. Thurston D. Brown and Mr. Alfred L. Finn have been engaged as research associates at the Bureau and have begun the referee testing of all materials exposed in the tests. These associates will appear on the records of the Bureau of Standards as employees of the National Research Council, for which purpose the Council has agreed to administer the funds for support of this work in a manner similar to the administration of many similar funds supporting research men at the Bureau. It has been anticipated that the referee testing program will require the employment of these two associates for a year. When this testing program has been completed and complete data have been obtained on the original materials exposed for test, a supplement to the present report will be published so that complete information on all materials exposed will be available in the publications of the Society.

With the publication of the data obtained in these referee tests at the Bureau of Standards, the work of the Wire Test Committee will have been completed. The inspection of exposed materials and the publication of inspection data and conclusions drawn from the tests were not the responsibility of the Wire Test Committee. These functions are the responsibility of a wire test inspection group, now being organized by Subcommittee VIII of Committee A-5, and it is to this group that the wire test program committee gladly turns over the further prosecution of the wire tests.

Respectfully submitted on behalf of the Wire Test Committee,

F. F. FARNSWORTH,
Chairman.

APPENDIX TO THE REPORT OF THE WIRE TEST COMMITTEE

The wire test program, as approved by Subcommittee VIII in 1934,[1] has been completed essentially as planned.

The following materials have been exposed in locations that are considered representative of the atmospheric conditions met by these products:

Farm-Field Fencing Barbed Wire
Unfabricated Wire Wire Strand
Chain-Link Fence

PLAN OF THE TEST

The primary purpose of the tests is to determine the effect of such factors as base-metal composition, gage, type of coating, weight of coating, etc., on the serviceability of zinc-coated strand, barbed, and woven wire fencing and unfabricated wire. All samples included in the tests have been carefully prepared and selected to represent hand-picked samples rather than the commercial run of product. Limits on the composition of base metal and the weight of coating and the classes of weights of coating have been very carefully chosen. No test materials have been purchased in the open market.

Only unfabricated wire samples will be returned to a central testing laboratory for determination of progressive changes in the physical properties of the wire as weathering continues. The fencing, barbed wire, and strand samples will be inspected at the test sites for general appearance, spread of rusting, etc.

The collecting, testing, marking and shipping of the samples to the test sites have been carried out so that the identity of the producer of a particular sample remains unknown. All records pertaining to the source of supply of test materials are to be destroyed as soon as data have been obtained on the original chemical and physical properties of the materials.

The committee has decided that no particular end point shall be sought in determining the serviceability of test specimens. It is felt that a rather complete picture of the deterioration of the samples, including extent of rusting, change in tensile properties and weight of coating, and decrease in cross-section of wires will furnish information suitable for predicting serviceability of the products.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 159 (1934).

TEST LOCATIONS

The test locations and the types of materials under test at each location are shown in Table I.

TABLE I.—TEST LOCATIONS.

The ✓ mark indicates that only those materials checked at each location have been exposed at that test site.

TEST LOCATIONS	FARM FENCE AND BARBED WIRE	UNFABRICATED WIRE	WIRE STRAND	CHAIN-LINK FENCE
State College, Pa.	**	✓*	✓	**
Lafayette, Ind.	✓	✓	✓	✓
College Station, Tex.	✓	✓	✓	..
Ames, Iowa	✓	✓	✓	..
Ithaca, N. Y.	✓	✓	✓	✓
Pittsburgh, Pa.	..	✓*	✓	✓
Sandy Hook, N. J.	..	✓*	✓	✓
Bridgeport, Conn.	..	✓	✓	✓
Manhattan, Kans.	✓	✓	✓	..
Davis, Calif.	✓	✓	✓	✓
Santa Cruz, Calif.	✓	✓	✓	✓

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* Additional samples have been provided at these test locations for determining the loss of weight of zinc coatings.

** Samples have not yet been erected.

A severe industrial atmosphere is obtained at the Pittsburgh, Pa., test site located at Brunot Island in the Ohio River about two miles west of the city of Pittsburgh. It is on the property of the Duquesne Light Co.

The site at Bridgeport, Conn., is also an industrial exposure, probably somewhat more moderate than the Pittsburgh location. The samples are exposed in the test yard of the American Chain Co.

An eastern seacoast exposure is obtained at Sandy Hook, N. J. The test field is located within the government reservation at Fort Hancock.

On the west coast, the University of California has provided a location at Santa Cruz which is essentially a seacoast exposure. It is located approximately three miles from the Pacific Ocean.

Rural exposures have been obtained at points widely distributed throughout the United States. These test fields have been obtained through the coöperation of the Agricultural Departments of the following colleges and Experiment Stations: Pennsylvania State College, State College, Pa.; Purdue University, Lafayette, Ind.; Texas Agricultural Experiment Station, College Station, Tex.; Iowa State College, Ames, Iowa; Cornell University, Ithaca, N. Y.; and Kansas State College, Manhattan, Kans. The location at Davis, Calif., also provided by the University of California, is inland approximately eighty miles and may be considered as a typical rural exposure for this section.

The test sites at Pittsburgh, Sandy Hook, and Pennsylvania State College adjoin the test racks on which the sheet tests of Subcommittee VIII are being made.

MATERIALS

Farm-Field Fencing:

Farm-field fencing, fabricated from wires of the following materials, are included in the tests:

- Uncoated copper-bearing steel
- Zinc-coated copper-bearing steel
- Corrosion-resistant steel
- Copper-covered steel
- Lead-coated steel

All farm fence samples with the exception of the lead-coated specimens are of standard hinge-joint type, 39 in. in height with 6-in. stay spacing.

The base metals of the wires composing the zinc-coated and uncoated fences were submitted as being basic open-hearth, copper-bearing steel containing 0.20 to 0.25 per cent copper and 0.08 to 0.20 per cent carbon.

The bare wire samples were bright lime-drawn and lead-annealed in such a manner as to produce physical properties approximating those of the zinc-coated samples. The wires were not coated with any substance after drawing.

Four types of farm-field fencing were exposed as shown in Table II.

TABLE II.—TYPES OF FARM-FIELD FENCING.

Type	Gage[a] of Top and Bottom Line Wire	Gage[a] of Intermediate Line Wires and Stay Wires	Number of Samples
1	No. 9	No. 9	17
2	No. 9	No. 11	19
3	No. 10	No. 12½	16
4	No. 11	No. 14½	15

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[a] Steel wire gage.

Classification of the samples of farm-field fencing as to types and weights of coating are shown in Table III.

TABLE III.—CLASSES OF ZINC-COATED FARM-FIELD FENCING AS TO TYPES AND WEIGHTS OF COATING.

Class	Type of Fence	Weight of Coating, oz. per sq. ft. of uncoated wire surface	Number of Samples Exposed
1 (Bare)	1, 2, 3 and 4	0.0	8
2	3 and 4	0.20 to 0.30	6
2	1 and 2	0.25 to 0.35	5
3	3 and 4	0.35 to 0.45	6
3	1 and 2	0.45 to 0.55	6
4	3 and 4	0.50 to 0.60	7
4	1 and 2	0.60 to 0.70	8
5	3 and 4	0.70 to 0.85	8
5	1 and 2	0.80 to 1.00	8
6	1 and 2	1.6 to 1.8	3
7	1 and 2	2.4 min.	2

Provision has been made for testing fencing made from the following alloys of corrosion-resistant steel:

18 per cent chromium, 8 per cent nickel, 0.12 per cent carbon (max.), cold drawn to approximately 300,000 lb. per sq. in. tensile strength.

12 to 14 per cent chromium, 0.12 per cent carbon (max.), air quenched, tempered to 1100 F. cold drawn to approximately 140,000 lb. per sq. in. tensile strength.

12 to 14 per cent chromium, 0.12 per cent carbon (max.), drawn to finished size then air quenched to approximately 175,000 lb. per sq. in. tensile strength.

TABLE IV.—SUMMARY OF SAMPLES OF FARM-FIELD FENCING.

ZINC-COATED SAMPLES.

CLASS	WEIGHT OF COATING, OZ. PER SQ. FT. OF UNCOATED WIRE SURFACE	NUMBER OF TYPES	NUMBER OF SAMPLES
1	bare	4	8
2	0.20 to 0.30 0.25 to 0.35	4	11
3	0.35 to 0.45 0.45 to 0.55	4	12
4	0.50 to 0.60 0.60 to 0.70	4	15
5	0.70 to 0.85 0.80 to 1.00	4	16
6	1.6 to 1.8	2	3
7	2.4 min.	2	2
		Total	67

SPECIAL SAMPLES.

MATERIAL	NUMBER OF TYPES	NUMBER OF SAMPLES
Corrosion-resistant steel, 18 per cent chromium, 8 per cent nickel	1	1
Corrosion-resistant steel, 12 to 14 per cent chromium (cold-drawn wire)	1	1
Corrosion-resistant steel, 12 to 14 per cent chromium (air-quenched wire)	1	1
Copper-covered steel	1	1
Lead-coated steel	1	1
	Total	5
Total Number of Samples		72

A sample of fencing made from each of these grades of corrosion-resistant steel has been exposed. The top and bottom line wires of the 18 per cent chromium, 8 per cent nickel fencing are No. 13 gage (tensile strength range 300,000 to 322,000 lb. per sq. in.); the intermediate line wires and stay wires are No. 15 gage (tensile strength approximately 265,000 lb. per sq. in.). In the 12 to 14 per cent chromium (cold-drawn wire), the top and bottom line wires and the intermediate line and stay

TABLE V.—UNFABRICATED WIRE.

BARE AND ZINC-COATED SAMPLES

Gage (Steel Wire Gage)	Bare		Weight of Coating, oz. per sq. ft. of uncoated wire surface										
	Non-Copper-Bearing	Copper-Bearing	0.20 to 0.30	0.25 to 0.35	0.35 to 0.45	0.45 to 0.55	0.50 to 0.60	0.60 to 0.70	0.70 to 0.85	0.80 to 1.00	1.2 to 1.5	1.6 to 1.8	2.4 min.
6	2	2	..	2	..	2	..	3	..	2	1	1	..
9	2	2	..	3	..	5	..	5	..	4	1	3	2
11	2	2	..	3	..	4	..	4	..	4	..	3	1
12½	2	2	3	..	3	..	3	..	5	..	..	2	..
14½	1	2	3	..	3	..	2	..	4	..	..	2	..
9[a]	..	..	..	..	..	..	..	..	..	1	..	..	..
12½[a]	..	..	..	..	..	..	..	..	1	..	..	..	..

Weight of Coating, oz. per sq. ft. of uncoated wire surface	Number of Gages	Number of Samples Exposed
Bare	5	19
0.20 to 0.30	2	6
0.25 to 0.35	3	8
0.35 to 0.45	2	6
0.45 to 0.55	3	11
0.50 to 0.60	2	5
0.60 to 0.70	3	12
0.70 to 0.85	2	9
0.80 to 1.00	3	10
1.2 to 1.5	2	2
1.6 to 1.8	5	11
2.4 min	2	3
Zinc coated and chromated	2	2
Total Number of Bare and Zinc-coated Samples		104

SPECIAL WIRE SAMPLES

Gage (Steel Wire Gage)	Corrosion-Resistant Steel		
	18 per cent Chromium, 8 per cent Nickel	12 to 14 per cent Chromium (cold-drawn wire)	12 to 14 per cent Chromium (air-quenched wire)
9	..	1	..
10	..	..	1
12	1	1	..
12½	..	..	1
14	1	..	..
15	1	..	..
17	..	1	..
18	..	..	1
20	1	..	..
	Total Number of Corrosion-resistant Steel Samples		10

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[a] Zinc-coated and chromated.

TABLE V (*Continued*).

Gage (Steel Wire Gage)	Copper-covered Steel	Lead-coated Steel
6	..	1
9	1	1
11	1	1
$14\frac{1}{2}$	..	1
	Total Number of Copper-covered Steel and Lead-coated Steel Samples............ 6	
Total Number of Samples of Unfabricated Wire...... 120		

wires are No. 12 gage. In the 12 to 14 per cent chromium (air-quenched wire), the top and bottom line wires and the intermediate line and stay wires are No. $12\frac{1}{2}$ gage.

NOTE.—The 16 to 18 per cent chromium, and 4 to 6 per cent chromium fencings, referred to in the Wire Test Program,[1] were not submitted.

One type of copper-covered steel fencing was erected in which the top and bottom line wires are No. 9 gage and the intermediate line wires and stay wires are No. 11 gage. The wire used in this fencing is in the "30 per cent conductivity" class. For the sizes of wire in the tests, the thickness of copper is given by the manufacturer as not less than 10 per cent of the radius of the wire.

There is also included in the tests one type of lead-coated steel fencing composed of wires having a minimum weight of coating of 1.5 oz. of lead per sq. ft. of bare wire surface. The top and bottom line wires and the intermediate line and stay wires of this fence are No. 9 gage.

Table IV shows the number of samples of farm-field fencing of all classes that have been erected.

Unfabricated Wire:

Unfabricated wire samples were furnished in the same materials as farm-field fencing. In addition to these materials bare wires made from non-copper-bearing, basic open-hearth steel, containing 0.08 to 0.20 per cent carbon, are included.

The zinc-coating classifications are the same as for farm-field fencing. The tests include Nos. 6, 9, 11, $12\frac{1}{2}$ and $14\frac{1}{2}$ gage wires.

The unfabricated wire tests also include zinc-coated wire which has been treated with a dichromate solution. These samples were galvanized by the usual hot-dip process and chromated by the New Jersey Zinc Co.

The classes of wire and the number of samples that have been erected are shown in Table V.

Barbed Wire:

All of the barbed wire samples are made from No. $12\frac{1}{2}$ gage line wires and $14\frac{1}{2}$ gage, 4-point barb hog wire. The base metal of the bare and zinc-coated barbed wire samples is the same as for farm-field fencing. Copper-covered steel and lead-coated steel barbed wire have also been exposed. The coating classifications and the number of samples exposed are shown in Table VI.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 172 (1934).

TABLE VI.—BARBED WIRE.

CLASS	WEIGHT OF COATING, OZ. PER SQ. FT. OF UNCOATED WIRE SURFACE	NUMBER OF SAMPLES
1	Bare	2
2	0.20 to 0.30	3
3	0.35 to 0.45	3
4	0.50 to 0.60	3
5	0.70 to 0.85	3
6[a]	0.85 to 1.0	3
7	Zinc-coated after, or coatings in excess of 1.6, min.	3
8	2.4 to 2.6	1
9	Copper-covered steel	1
10	Lead-coated steel	1
Total Number of Samples		23

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[a] One sample in this class was furnished with aluminum barbs.

Wire Strand:

All samples of strand are $\frac{3}{8}$ in. in diameter and are composed of seven wires, each 0.120 in. in diameter. The base metal is of the grade known as Siemens-Martin. Table VII shows coating classifications and number of samples exposed.

TABLE VII.—WIRE STRAND.

CLASS	WEIGHT OF COATING, OZ. PER SQ. FT. OF UNCOATED WIRE SURFACE	NUMBER OF SAMPLES
1	0.80 to 0.95	4
2	1.0 to 1.2	3
3	1.3 to 1.5	3
4	1.6 to 1.8	1
5	2.4 to 2.6	1
6	Lead-coated steel	1
Total Number of Samples		13

Chain-Link Fence:

Chain-link fence has been submitted, made from copper-bearing steel galvanized before weaving and also galvanized after weaving. Corrosion-resistant steel chain-link fencing of the 18 per cent chromium, 8 per cent nickel grade, copper-covered steel and lead-coated steel fencings are also included. The weights of coating, gage sizes and number of samples exposed are shown in Table VIII.

TABLE VIII.—CHAIN-LINK FENCE.

ZINC-COATED SAMPLES

GAGE (STEEL WIRE GAGE)	GALVANIZED BEFORE OR AFTER WEAVING	WEIGHT OF COATING, OZ. PER SQ. FT. OF UNCOATED WIRE SURFACE	NUMBER OF SAMPLES
9	Before	under 0.50	3
9	After	under 0.50	1
9	Before	1.20 to 1.50	1
9	After	1.20 to 1.50	5
6	Before	1.20 to 1.50	1
6	After	1.20 to 1.50	4
9	Before	2.4 to 3.0	2
9	After	2.4 to 3.0	1

SPECIAL SAMPLES

GAGE (STEEL WIRE GAGE)	MATERIAL	NUMBER OF SAMPLES
14	Corrosion-resistant steel	1
0.128 in. Diam	Copper-covered steel	1
9	Lead-coated steel	1
Total Number of Samples		21

PREPARATION OF TEST SAMPLES

A suitable building (37-18 Tenth St., Long Island City, N. Y.), was secured for the reception and fabrication of the samples. Great care was observed to avoid injuring them. They always rested on either burlap or heavy building paper, never coming in contact with the floor, or being dragged over it.

Due to the prevailing commercial conditions, many of the deliveries of samples to the shop were much delayed, which markedly increased the cost of assembly and fabrication, and delayed erection in the field.

In handling the material in the shop, as a safeguard against confusion of identity, only one sample was opened at a time. The manufacturers' marks were all removed and the code designation applied, a record of the same being made in a book. Immediately, the required number of specimens was cut off and each marked with the code designation. These marks, for unfabricated wires to be mounted on frames, were on tough manila tags having a hole at each end, through both of which the wire was threaded. The tag remained on each wire throughout all shop operations and was removed only when the frame was about to be packed for shipment. These wires are identified by position on the numbered frame, and reference to the appended list. To all specimens other than unfabricated wire, the final metallic field tags were at once applied.

A system of designating marks was arranged for the samples and for the individual specimens cut from each. Each specimen mark consists of two letters, followed by three numerals, for example, MA236. The first letter indicates the test field at which the individual specimen is exposed; the second letter indicates the kind of material, for example farm fence, barbed wire, etc. The first numeral indicates the gage of the wire. In the case of farm fence, the gage used is that of the intermediate line wires and the stay wires, the gage of these wires not being duplicated in any two classes of fence. The series formed by the last two numerals, 00 to 99 inclusive, was divided into suitable groups to represent the nominal weights of coating of zinc-coated wires, including the two kinds of carbon-steel wire with zero coating. Special numerals were applied to the corrosion-resistant steel, lead-coated steel and copper-covered steel. The marks also indicate the side of the rack on which the specimens of strand and unfabricated wire are placed.

Full details of the individual markings are appended.[1]

The markings above described were stamped plainly on metallic tags, two of which were attached to each frame of plain wire and two to each specimen of other material. Zinc-coated and uncoated carbon-steel materials were marked with solid zinc tags, attached at both ends by heavy solid zinc wires. Similarly, copper-covered steel was marked with copper tags attached by copper wires, lead-coated steel by lead tags attached by lead wires, and corrosion-resistant steel by corrosion-resistant steel tags attached by corrosion-resistant steel wires.

Farm fencing was cut to length and the specimens joined into the fences by bringing together the adjacent end stay wires, then splicing all the line wires across the two stay wires.

The unfabricated wires were applied to the frames as specified by the 1934 report,[2] by electric spot welding, while the wires were held in position in a jig. The zinc-coated wires required no surface preparation. The uncoated carbon-steel wires were first cleaned free from mill scale at the point of welding, with emery cloth. Copper-covered and lead-coated steel wires were successfully welded to the frames without preparation. It was found impracticable, with available equipment, to spot weld the corrosion-resistant steel wires to the frames. Short galvanized loops were therefore first welded to the frame and the ends of the corrosion-resistant steel specimens looped through. After welding, the joints of all wires were cleaned of dross with a stiff brush, then liberally coated with a thick paint composed of metallic zinc dust in a vehicle having a bakelite base. For additional protection of the welds of copper-covered and lead-coated steel wires, to avoid electrolysis, the joints and adjacent part of the frame were tightly wrapped with a specially durable quality of insulating tape, while the paint was still soft. A second thick coat of paint was then applied over the tape.

Barbed wire specimens were not joined in the shop, but the ends were individually looped and twisted around the post arms.

Specimens of strand were very successfully strung individually in the field by means of strand clamps.

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[1] See pp. 140 to 150.

[2] *Loc. cit.*

When possible, adjacent sections of chain-link fencing were joined by threading a picket through both, as in the ordinary erection of fences. Often, however, the two specimens did not exactly match in mesh or in direction of coiling, and other means were necessary. The most satisfactory was a pair of straight No. 6 galvanized wires threaded vertically through both adjacent ends.

For shipment to the nearby test fields, farm fence was rolled up and wrapped in waterproofed burlap. For the more distant test locations, the rolls of farm fence were wrapped in burlap and then crated. All barbed wire specimens were coiled and boxed. The smaller parcels of chain-link fence were wrapped in burlap, and sewed. The larger parcels were wrapped in burlap and then crated. The specimens of strand were laid out as nearly straight as possible, tied together at frequent intervals and then spirally wrapped in burlap. The frames of unfabricated wire were packed in boxes with separating slats so arranged that the welds did not touch the adjoining frames.

WEIGHT LOSS DETERMINATIONS

Other specimens of unfabricated wires have been exposed for the determination of loss of weight of the zinc coating. This is a feature not included in the report of 1934, but was subsequently requested by several members including the National Bureau of Standards. For this purpose, there were used wires of three gages coated by each of several processes. Duplicate specimens of each of these samples were placed on each frame and four frames each were placed on the East side and on the West side of the rack. Before mounting these specimens on the frames, precision measurements were made at the Bureau of Standards. For this purpose, ends of the specimens were smoothly faced off in a lathe and measured within 0.001 in. They were then weighed within a milligram by suspending them vertically from one pan of a laboratory fine balance. The balance case was provided with a well under one of the pans, deep enough to accommodate the specimen. Four periodic tests of loss of weight will be made. Weight test specimens are exposed at Sandy Hook (sea air), Pennsylvania State College (pure mountain air) and Pittsburgh (industrial air).

Welding the wires to the frames obviously changes their weight and they cannot be removed without further change in weight at the two ends where the welds are located. For a precision determination of the loss of weight, it is not permissible to compare the weight of one specimen with the weight of another from the same coil. By cutting the minimum amount from the ends of the exposed wire to remove only the portion injured by the welding and painting, there will still remain 90 to 95 per cent of the same wire that was originally measured and weighed. This remainder will be measured and weighed as before, after suitable cleaning. The weights per unit of length, before and after weighing, will thus be comparable and will be a reliable basis for determining the loss of weight per unit of surface. A list of the samples giving their positions on the frames is appended.[1]

ERECTION

Shipments of samples to the test fields were completed during October, 1936. Meanwhile, the supporting post and rack material had been secured and shipped to the test fields, in order that it might be erected and made ready for the reception of the test samples.

For the lighter farm fences, Nos 1 to 5, the end posts are tubular, 2 in. in outer diameter and the wall thickness of No. 8 gage. The intermediate line posts are of T section, $1\frac{3}{8}$ in. by $1\frac{3}{8}$ in., the webs being $\frac{7}{64}$ in. in thickness. Fences Nos. 6, 7 and 8 contain the more durable samples. The end posts are $2\frac{1}{2}$ in. in outer diameter and of No. 8 gage wall thickness. The intermediate line posts are of U section, $1\frac{3}{16}$ in. by $2\frac{3}{16}$ in., the webs being $\frac{11}{64}$ in. in thickness. The end posts and intermediate dividing posts of chain-link fences 9 and 10 are $2\frac{1}{2}$ in. by No. 8. The chain link intermediate line posts are 2 in. by No. 8. All fence posts and pipe supports are heavily galvanized with the exception of the posts used with the lighter farm fences Nos. 1 to 5. These posts are heavily painted.

All end posts are provided with substantial diagonal tubular braces. All end posts and braces of farm fence are set in concrete; line posts are driven. For the chain-link fences, all braces and all posts, both end and line, are set in concrete. All tubular posts for either kind of fence are filled with concrete to prevent internal corrosion. Chain-link fences are provided with top rails consisting of $1\frac{1}{4}$-in. ($1\frac{5}{8}$-in. outside diameter) standard-weight galvanized pipe.

In the arrangement and erection of supports and test samples in the test fields, the drawings shown in the 1934 report were followed quite closely. A minor change was made in making the strand attachments. It was found desirable to employ two-bolt and three-bolt strand clamps as a means of supporting the strand at the crossarms. Ten lines of fence were sufficient instead of eleven. Eight were used for farm fence and two for chain-link fence.

It seemed undesirable, from cost considerations, to erect each short-fence specimen individually, even though this would result in a minimum of repair maintenance at the test fields as failures occurred. Consequently, ten samples were fabricated into a test fence and erected as a unit. In selecting appropriate specimens for each test fence, consideration was given to grouping of the samples. For example, lightest gage, lightest weight coating specimens were kept separate from heavy gage, heavy weight coating specimens.

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[1] See List III, p. 148.

The arrangement of the fences in the field and the location of each specimen of farm fence, chain-link fence and of barbed wire, are shown in Fig. 1. The fence fabric was applied to the posts by the usual method. The fabric was leaned up against the posts in appoximately its final position and held loosely by temporary wire ties. For farm fence, one end was attached to its end post by wrapping all the line wires tightly around the post then tightly around themselves. The usual stretching tools were employed. Clamping bars were attached near the other end and the fabric stretched by means of pulleys or winches. The line wires were then attached to the second end post as above. For the chain-link fence, the process was similar except that the fabric was attached to the end posts by the usual end tension rods and post collars whose lugs held the tension rods by bolts. Both kinds of fence were permanently attached to each line post by five ties of heavily galvanized wire. The chain link was also attached to the top rail by five ties in each section between posts. Specimens of copper-covered steel fence were cut a little shorter than the distance between posts and connected to the posts by means of heavy galvanized wires with an intermediate strain insulator of porcelain on each wire.

The barbed wire is stretched on post arms and over farm fence having the same weight of coating. To increase the length of sample exposed, two 16-ft. lengths of each sample have been installed at each test site.

To support one complete set of unfabricated wire specimens, five frames such as are described in the 1934 report, were required. The specimens are distributed among the five frames in the order of relative durability, on a basis similar to that described above for fencing. Seven such sets are exposed at each test field, permitting seven periodic tension tests. The specimens are of such length as to provide tests in triplicate. The distribution of each set among five frames also permits tests to be made of the less and of the more durable samples at different times from each field, as may be determined by the condition of the samples.

NOTE.—At Sandy Hook, Pennsylvania State College, and Brunot Island, space available on the West side of the racks was not sufficient for all of the tension test frames. The remaining frames were consequently mounted on available spaces on the existing hardware exposure racks or (at Pennsylvania State) on a separate temporary rack. These specially mounted frames should, in their respective classes, be the first to be removed for periodic tests.

The dates when the erection was completed and the exposure begun at the individual fields are as follows:

Bridgeport, Conn.	November 14, 1936
(except three samples of corrosion-resistant steel farm fence, erected in January, 1937)	
Sandy Hook (Fort Hancock), N. J.	November 17, 1936
State College, Pa.	January 9, 1937 (Wire and Strand)
Brunot Island, Pittsburgh, Pa.	November 5, 1936

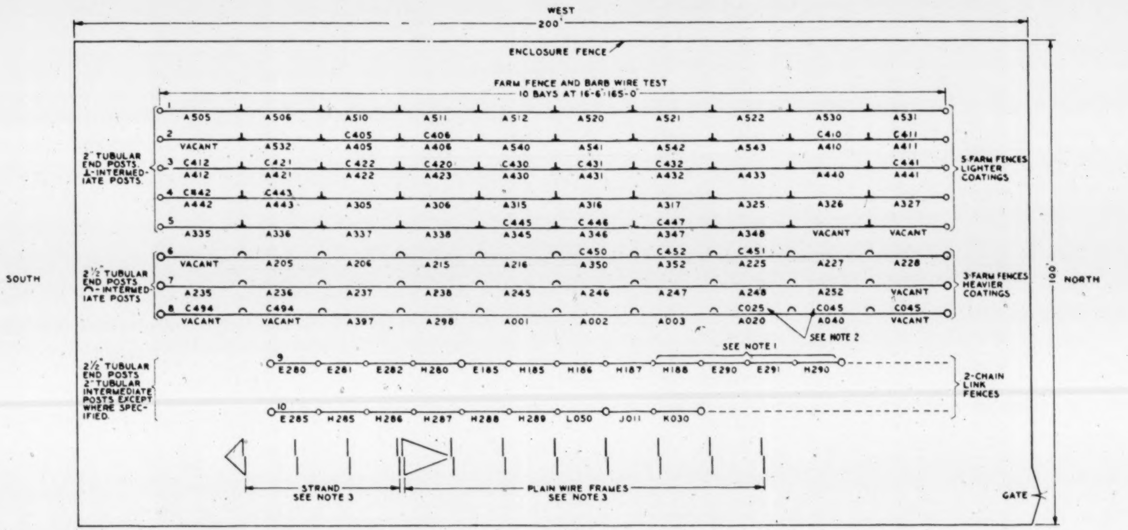
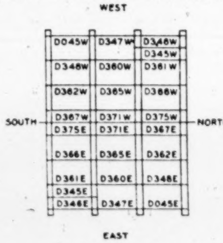

WEST
200'
ENCLOSURE FENCE
FARM FENCE AND BARB WIRE TEST
10 BAYS AT 16'-6" 165'-0"
SOUTH
NORTH
100'
GATE
2" TUBULAR END POSTS ⊥-INTERMEDIATE POSTS
2½" TUBULAR END POSTS ⌒-INTERMEDIATE POSTS
2½" TUBULAR END POSTS 2" TUBULAR INTERMEDIATE POSTS EXCEPT WHERE SPECIFIED
5 FARM FENCES LIGHTER COATINGS
3 FARM FENCES HEAVIER COATINGS
2-CHAIN LINK FENCES
SEE NOTE 1
SEE NOTE 2
STRAND SEE NOTE 3
PLAIN WIRE FRAMES SEE NOTE 3
EAST

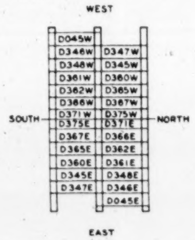
FIG 1

NOTES

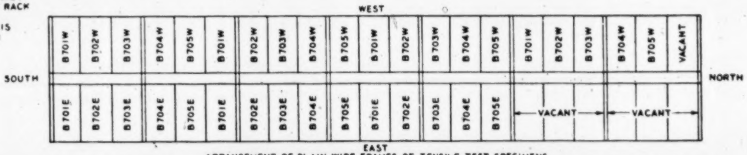
1. AT ITHACA FIELD THESE FOUR SAMPLES ARE IN REVERSE ORDER, I.E -H290, E291, E290, H188.
2. DUE TO LOCAL VARIATIONS IN SETTING POSTS THE LOCATION OF SAMPLES C025 AND C045 IS SOMEWHAT VARIED BUT ALWAYS AT OR NEAR THE NORTH ENDS OF FENCES 7 OR 8
3. DETAILS FOR MOUNTING OF STRAND AND PLAIN WIRE FRAMES ARE SHOWN IN FIGS 2, 3, 4 & 5
4. THERE ARE NO WEIGHT TEST SPECIMENS AT THESE FIELDS.
5. THESE THREE TENSILE TEST FRAMES ARE VARIOUSLY PLACED ON THE HARDWARE TEST RACKS AT SANDY HOOK AND PITTSBURGH AND ON AN AUXILIARY RACK AT PENN. STATE COLLEGE.
6. AT PITTSBURGH THE ORDER OF THE FRAMES IS REVERSED, I.E. THE NUMBERS PROGRESS FROM NORTH TO SOUTH


WEST
SOUTH
NORTH
EAST

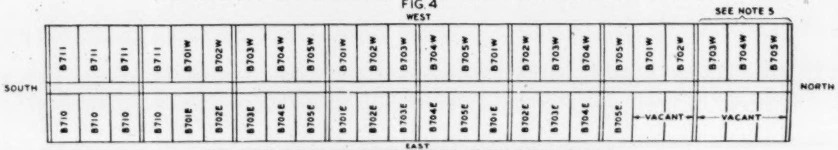
ARRANGEMENT OF STRAND SPECIMENS ON RACKS AT BRIDGEPORT, ITHACA, LAFAYETTE, AMES, MANHATTAN, COLLEGE STATION TEXAS, DAVIS AND SANTA CRUZ.
FIG. 2


WEST
SOUTH
NORTH
EAST

ARRANGEMENT OF STRAND SPECIMENS ON RACKS AT SANDY HOOK, PENN. STATE COLLEGE AND PITTSBURGH
FIG. 3


WEST
SOUTH
NORTH
VACANT
EAST

ARRANGEMENT OF PLAIN WIRE FRAMES OF TENSILE TEST SPECIMENS AT BRIDGEPORT, ITHACA, LAFAYETTE, AMES, MANHATTAN, COLLEGE STATION TEXAS, DAVIS AND SANTA CRUZ. (SEE NOTE 4)
FIG. 4


WEST
SEE NOTE 5
SOUTH
NORTH
VACANT
EAST

ARRANGEMENT OF PLAIN WIRE FRAMES OF TENSILE AND WEIGHT TEST SPECIMENS AT SANDY HOOK, PENN. STATE COLLEGE AND PITTSBURGH (SEE NOTE 6).
FIG 5

PLATE I.—Diagrams Showing Location of the Individual Specimens in the Test Racks at the Eleven Test Locations.

Ithaca, N. Y	November 12, 1936
Lafayette, Ind	October 31, 1936
Ames, Iowa	November 28, 1936
Manhattan, Kans	December 4, 1936
College Station, Tex	December 26, 1936
Davis, Calif	December 12, 1936
Santa Cruz, Calif	December 18, 1936

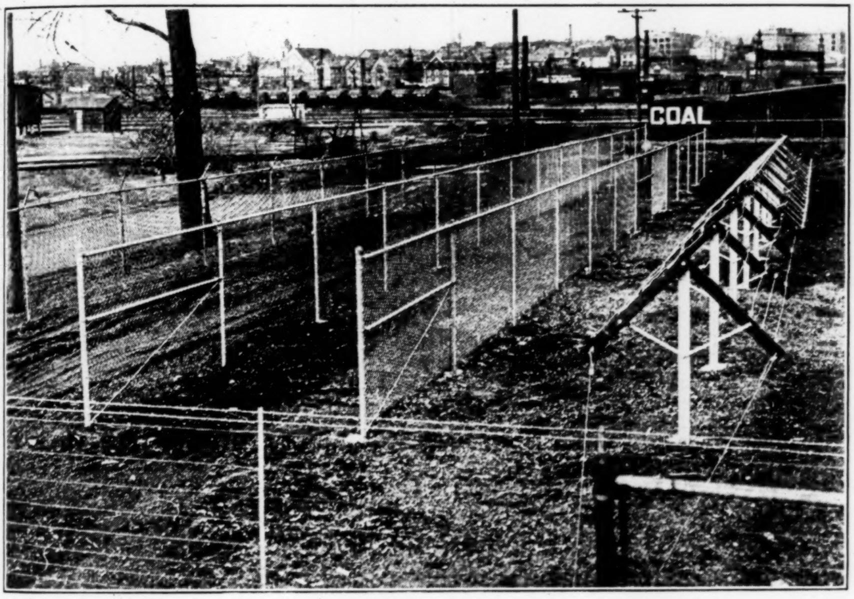


FIG. 6.—Test Field at Bridgeport, Conn.

The location of the individual specimens in the test fields is shown in the accompanying Plate I, as follows:

Fig. 1	Farm-field fencing, chain-link fence, barbed wire
Figs. 2 and 3	Wire strand
Fig. 4	Unfabricated wire frames at fields having tension test frames only
Fig. 5	Unfabricated wire frames at fields having also weight test specimens

The locations of specimens are the same at all fields except as noted on the figures.

The accompanying photographs in Figs. 6 to 12 illustrate the general assembly of test specimens at each test site and details of construction and mounting of the individual specimens.

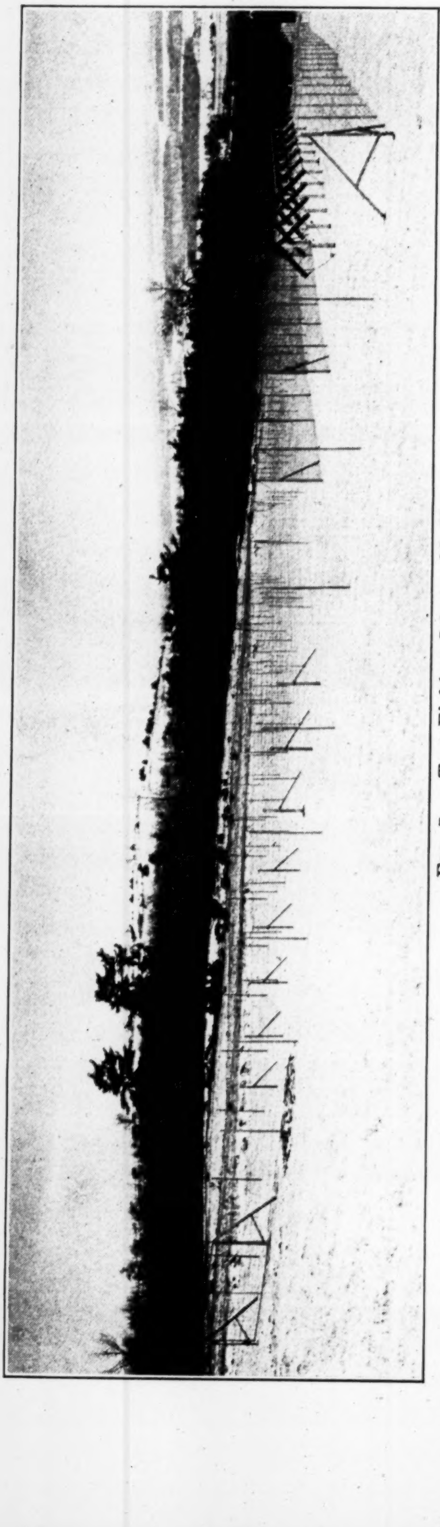


FIG. 7.— Test Field at Ithaca, N. Y.

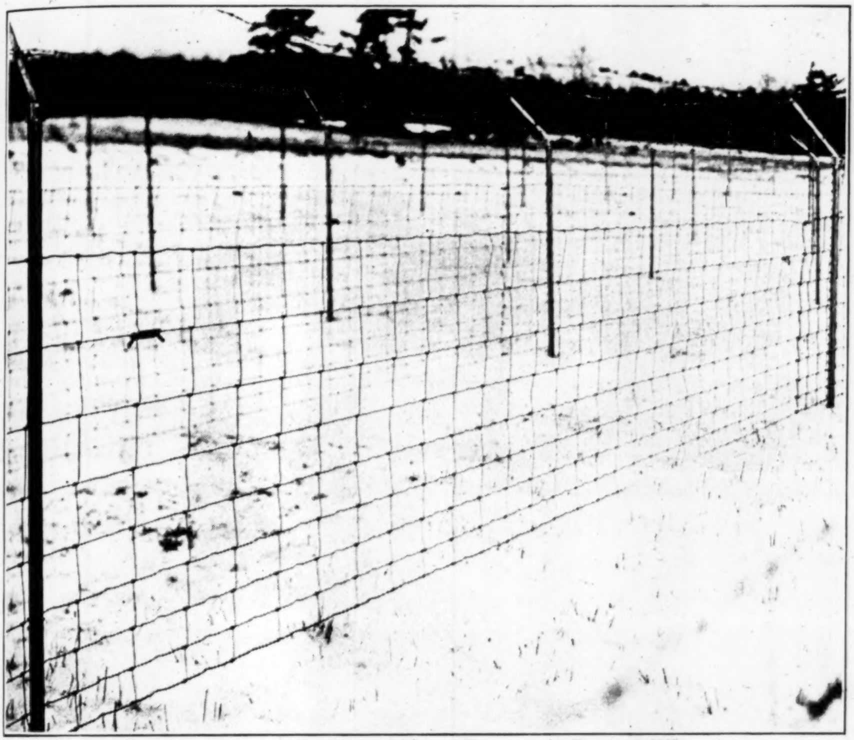


FIG. 8.—Typical Section of Farm Fence and Barbed Wire.

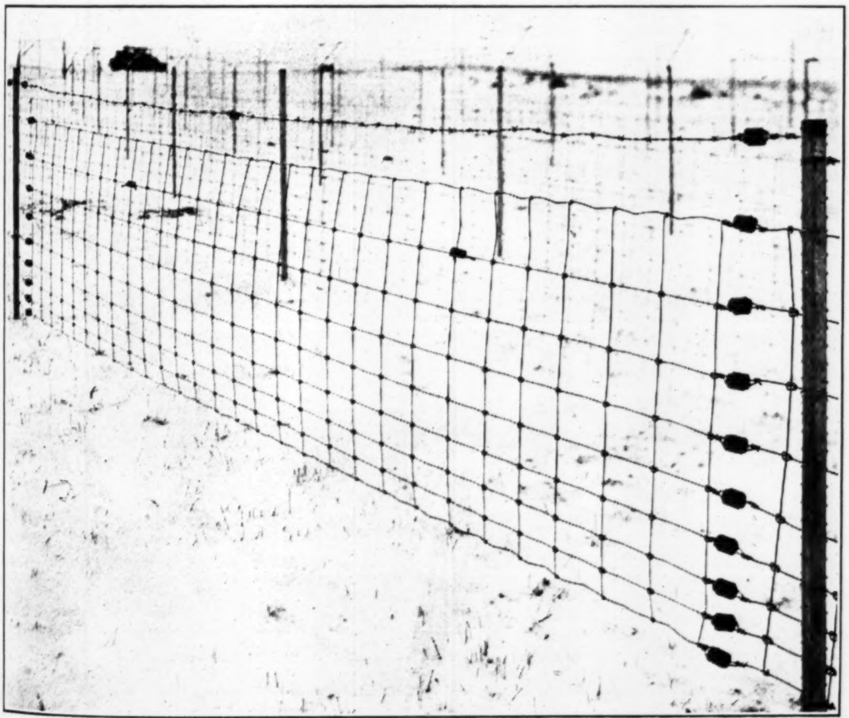


FIG. 9.—Special Fencing Sections Showing Insulators Between Dissimilar Metals.

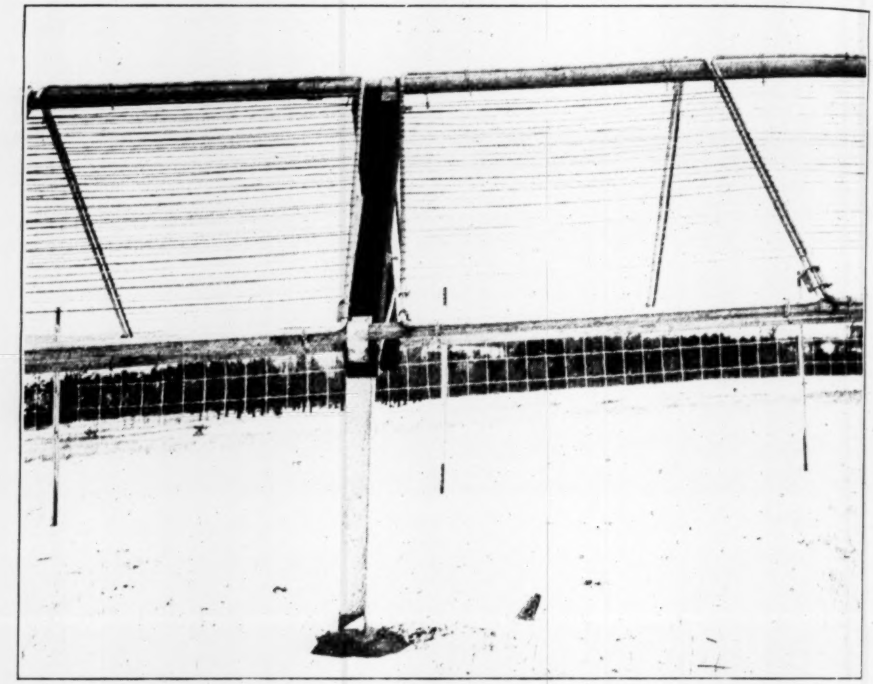


FIG. 10.—Typical Installation of Frames of Unfabricated Wire.

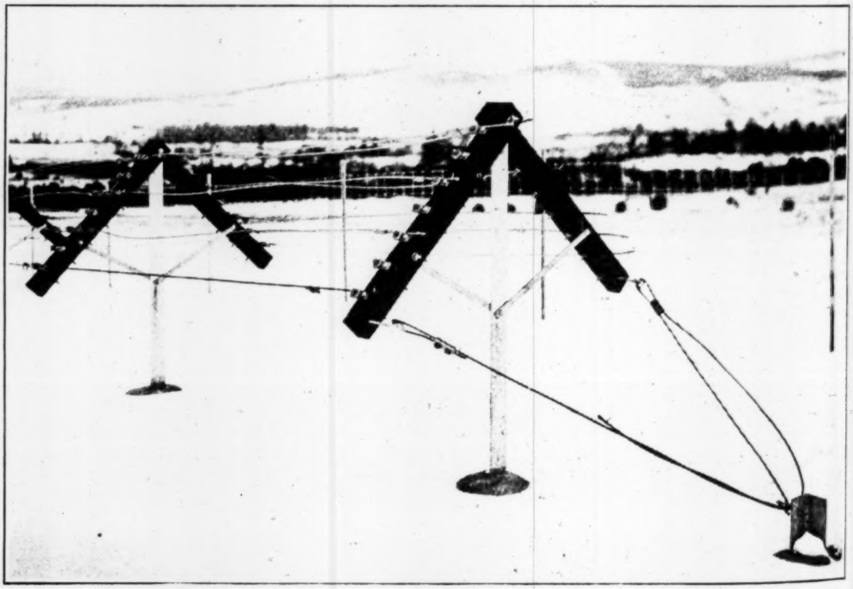


FIG. 11.—Details of Erection of Wire Strand Samples.

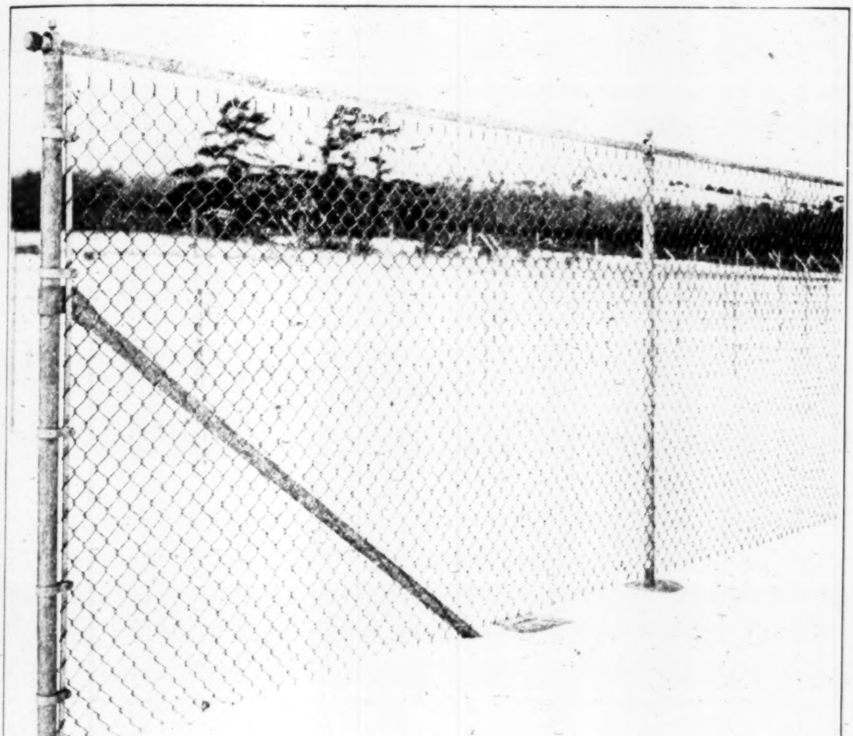


FIG. 12.—Chain-Link Fence Construction.

LIST OF INDIVIDUAL SAMPLES AND SYSTEM OF MARKING

SYSTEM OF MARKING:

Test Fields:

A — Bridgeport, Conn.
B — Sandy Hook, N. J.
C — Ithaca, N. Y.
D — State College, Pa.
E — Pittsburgh, Pa.
H — Lafayette, Ind.
J — Ames, Iowa
K — Manhattan, Kans.
L — College Station, Tex.
M — Davis, Calif.
O — Santa Cruz, Calif.

Kinds of Material:

A — Farm-Field Fencing
B — Unfabricated Wires
C — Barbed Wire
D — Wire Strand
E — Chain-Link Fence, Galvanized Before Weaving
H — Chain-Link Fence, Galvanized After Weaving
J — Chain-Link Fence, Corrosion-resistant Steel
K — Chain-Link Fence, Copper-covered Steel
L — Chain-Link Fence, Lead-coated Steel

Gage of Wire:

100 series	No. 6 gage
200 series	No. 9 gage
300 series	No. 11 gage
400 series	No. 12½ gage
500 series	No. 14½ gage

Numerical Designation of Special Materials:

000–015 series	Corrosion-resistant steel
020–030 series	Copper-covered steel
040–050 series	Lead-coated steel

Numerical Designation of Frames of Unfabricated Wire:

700–705 series	Frames of Unfabricated Wire for Tension Tests
710–711 series	Frames of Unfabricated Wire for Loss of Weight Tests

NOTE.—In addition, two samples of copper-bearing unfabricated wire, No. 6 and No. 9 gage, having a coating weight of 1.2 to 1.5 oz. per sq. ft. are designated B800 and B801.

Weight of Coating Classification:

00–04 series—Uncoated, non-copper-bearing steel
05–09 series—Uncoated, copper-bearing steel
10–14 series—0.20 to 0.30 oz. per sq. ft.[a]
15–19 series—0.25 to 0.35 oz. per sq. ft.[a]
20–24 series—0.35 to 0.45 oz. per sq. ft.[a]
25–29 series—0.45 to 0.55 oz. per sq. ft.[a]
30–34 series—0.50 to 0.60 oz. per sq. ft.[a]
35–39 series—0.60 to 0.70 oz. per sq. ft.[a]
40–44 series—0.70 to 0.85 oz. per sq. ft.[a]
45–49 series
  0.80 to 1.00 oz. per sq. ft.[a] Farm Fence and Unfabricated Wire
  0.85 to 1.00 oz. per sq. ft.[a] Barbed Wire
  0.80 to 0.95 oz. per sq. ft.[a] Wire Strand
50–59 series—1.6 to 1.8 oz. per sq. ft.[a]
60–64 series—1.0 to 1.2 oz. per sq. ft.[a]
65–69 series—1.3 to 1.5 oz. per sq. ft.[a]
70–74 series—1.6 to 1.8 oz. per sq. ft.[a]
75–79 series—2.4 to 2.6 oz. per sq. ft.[a]
80–84 series—under 0.50 oz. per sq. ft.[a]
85–89 series—1.2 to 1.5 oz. per sq. ft.[a]
90–94 series—2.4 to 3.0 oz. per sq. ft.[a]
95–96 series—Chromated after zinc coating, unfabricated wire[a]
97–99 series—Extra-heavy coatings[a]

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[a] All zinc coatings are on copper-bearing steel.

LIST I.—FARM-FIELD FENCING.

(Arranged in same order as in the fences, South to North)

Fence Number	Sample Designation	Material	Gage (Steel Wire Gage)		Coating Classification
			Top and Bottom Line Wires	Intermediate Line Wires and Stay Wires	Weight of Coating, oz. per sq. ft. of uncoated wire surface
1	A505	Copper-bearing steel	11	14½	Bare
	A506	" "	11	14½	Bare
	A510	Zinc-coated, copper-bearing steel	11	14½	0.20 to 0.30
	A511	" " "	11	14½	0.20 to 0.30
	A512	" " "	11	14½	0.20 to 0.30
	A520	" " "	11	14½	0.35 to 0.45
	A521	" " "	11	14½	0.35 to 0.45
	A522	" " "	11	14½	0.35 to 0.45
	A530	" "	11	14½	0.50 to 0.60
	A531	" " "	11	14½	0.50 to 0.60
2	Vacant				
	A532	Zinc-coated, copper-bearing steel	11	14½	0.50 to 0.60
	A405	Copper-bearing steel	10	12½	Bare
	A406	" "	10	12½	Bare
	A540	Zinc-coated, copper-bearing steel	11	14½	0.70 to 0.85
	A541	" " "	11	14½	0.70 to 0.85
	A542	" " "	11	14½	0.70 to 0.85
	A543	" " "	11	14½	0.70 to 0.85
	A410	" " "	10	12½	0.20 to 0.30
	A411	" " "	10	12½	0.20 to 0.30
3	A412	Zinc-coated, copper-bearing steel	10	12½	0.20 to 0.30
	A421	" " "	10	12½	0.35 to 0.45
	A422	" " "	10	12½	0.35 to 0.45
	A423	" " "	10	12½	0.35 to 0.45
	A430	" " "	10	12½	0.50 to 0.60
	A431	" " "	10	12½	0.50 to 0.60
	A432	" " "	10	12½	0.50 to 0.60
	A433	" " "	10	12½	0.50 to 0.60
	A440	" " "	10	12½	0.70 to 0.85
	A441	" " "	10	12½	0.70 to 0.85
4	A442	Zinc-coated, copper-bearing steel	10	12½	0.70 to 0.85
	A443	" " "	10	12½	0.70 to 0.85
	A305	Copper-bearing steel	9	11	Bare
	A306	" "	9	11	Bare
	A315	Zinc-coated, copper-bearing steel	9	11	0.25 to 0.35
	A316	" " "	9	11	0.25 to 0.35
	A317	" " "	9	11	0.25 to 0.35
	A325	" " "	9	11	0.45 to 0.55
	A326	" " "	9	11	0.45 to 0.55
	A327	" " "	9	11	0.45 to 0.55

LIST I.—FARM-FIELD FENCING (*Continued*).

(Arranged in same order as in the fences, South to North)

Fence Number	Sample Designation	Material	Gage (Steel Wire Gage)		Coating Classification
			Top and Bottom Line Wires	Intermediate Line Wires and Stay Wires	Weight of Coating, oz. per sq. ft. of uncoated wire surface
5	A335	Zinc-coated, copper-bearing steel	9	11	0.60 to 0.70
	A336	" " "	9	11	0.60 to 0.70
	A337	" " "	9	11	0.60 to 0.70
	A338	" " "	9	11	0.60 to 0.70
	A345	" " "	9	11	0.80 to 1.00
	A346	" " "	9	11	0.80 to 1.00
	A347	" " "	9	11	0.80 to 1.00
	A348	" " "	9	11	0.80 to 1.00
	Vacant				
	Vacant				
6	Vacant				
	A205	Copper-bearing steel	9	9	Bare
	A206	" "	9	9	Bare
	A215	Zinc-coated, copper-bearing steel	9	9	0.25 to 0.35
	A216	" " "	9	9	0.25 to 0.35
	A350	" " "	9	11	1.6 to 1.8
	A352	" " "	9	11	1.6 to 1.8
	A225	" " "	9	9	0.45 to 0.55
	A227	" " "	9	9	0.45 to 0.55
	A228	" " "	9	9	0.45 to 0.55
7	A235	Zinc-coated, copper-bearing steel	9	9	0.60 to 0.70
	A236	" " "	9	9	0.60 to 0.70
	A237	" " "	9	9	0.60 to 0.70
	A238	" " "	9	9	0.60 to 0.70
	A245	" " "	9	9	0.80 to 1.00
	A246	" " "	9	9	0.80 to 1.00
	A247	" " "	9	9	0.80 to 1.00
	A248	" " "	9	9	0.80 to 1.00
	A252	" " "	9	9	1.6 to 1.8
	Vacant				
8	Vacant				
	Vacant				
	A397	Zinc-coated, copper-bearing steel	9	11	2.4 min.
	A298	" " "	9	9	2.4 min.
	A001	Corrosion-resistant steel, 12 to 14 per cent chromium, cold-drawn wire	12	12	......
	A002	Corrosion-resistant steel, 12 to 14 per cent chromium, air-quenched wire	12	12½	.....
	A003	Corrosion-resistant steel, 18 per cent chromium, 8 per cent nickel	13	15	....
	A020	Copper-covered steel	9	11	......
	A040	Lead-coated steel	9	9	......
	Vacant				

LIST II.—UNFABRICATED WIRES FOR TENSION TESTS.

The three numerals, representing the individual samples of plain wire, are the same as the three numerals representing the corresponding individual sample of farm fence, made from the same lot of wire.

In the following tabulations, the samples are listed in the same order as the samples on the exposure frames on which they are mounted, counting from top to bottom. The top of the frame is distinguished by the weld in the frame. The zinc number tags are on the bottom of the frame.

There are four identical sets of frames for tension tests, exposed on the West side of the racks. The numbers on these frames bear a suffix "W." There are three sets of these frames on the East side of the racks and these are distinguished by the suffix "E."

FRAME NO. 701, TENSION TESTS

Position of Wire on Frame	Sample Designation	Material	Gage (Steel Wire Gage)	Coating Classification
				Weight of Coating, oz. per sq. ft. of uncoated wire surface
1	Vacant			
2	B501	Non-copper-bearing steel	14½	Bare
3	B507	Copper-bearing steel	14½	Bare
4	B506	" " "	14½	Bare
5	B510	Zinc-coated, copper-bearing steel	14½	0.20 to 0.30
6	B511	" " "	14½	0.20 to 0.30
7	B512	" " "	14½	0.20 to 0.30
8	B520	" " "	14½	0.35 to 0.45
9	B521	" " "	14½	0.35 to 0.45
10	B522	" " "	14½	0.35 to 0.45
	Extra space between wires here			
11	B400	Non-copper-bearing steel	12½	Bare
12	B401	" "	12½	Bare
13	B411	Zinc-coated, copper-bearing steel	12½	0.20 to 0.30
14	B531	" " "	14½	0.50 to 0.60
15	B530	" " "	14½	0.50 to 0.60
16	B405	Copper-bearing steel	12½	Bare
17	B406	" "	12½	Bare
18	B540	Zinc-coated, copper-bearing steel	14½	0.70 to 0.85
19	B541	" " "	14½	0.70 to 0.85
20	B542	" " "	14½	0.70 to 0.85
	Extra space between wires here			
21	B543	Zinc-coated, copper-bearing steel	14½	0.70 to 0.85
22	B410	" " "	12½	0.20 to 0.30
23	Vacant			
24	B412	" " "	12½	0.20 to 0.30
25	B423	" " "	12½	0.35 to 0.45
26	B421	" " "	12½	0.35 to 0.45
27	B422	" " "	12½	0.35 to 0.45
28	Vacant			
29	Vacant			
30	Vacant			

LIST II.—UNFABRICATED WIRES FOR TENSION TESTS (*Continued*).

FRAME NO. 702, TENSION TESTS

Position of Wire on Frame	Sample Designation	Material	Gage (Steel Wire Gage)	Coating Classification
				Weight of Coating, oz. per sq. ft. of uncoated wire surface
1	B430	Zinc-coated, copper-bearing steel	12½	0.50 to 0.60
2	B300	Non-copper-bearing steel	11	Bare
3	B441	Zinc-coated, copper-bearing steel	12½	0.70 to 0.85
4	B443	" " "	12½	0.70 to 0.85
5	B305	Copper-bearing steel	11	Bare
6	B306	" "	11	Bare
7	B315	Zinc-coated, copper-bearing steel	11	0.25 to 0.35
8	B316	" " "	11	0.25 to 0.35
9	B325	" " "	11	0.45 to 0.55
10	B327	" " "	11	0.45 to 0.55
	Extra space between wires here			
11	B329	Zinc-coated, copper-bearing steel	11	0.45 to 0.55
12	B335	" " "	11	0.60 to 0.70
13	B336	" " "	11	0.60 to 0.70
14	B431	" " "	12½	0.50 to 0.60
15	B432	" " "	12½	0.50 to 0.60
16	B302	Non-copper-bearing steel	11	Bare
17	B440	Zinc-coated, copper-bearing steel	12½	0.70 to 0.85
18	B444	" " "	12½	0.70 to 0.85
19	B551	" " "	14½	1.6 to 1.8
20	B337	" " "	11	0.60 to 0.70
	Extra space between wires here			
21	B338	Zinc-coated, copper-bearing steel	11	0.60 to 0.70
22	B495[a]	" " "	12½	0.70 to 0.85 chromated
23	B450	" " "	12½	1.6 to 1.8
24	B442	" " "	12½	0.70 to 0.85
25	B451	" " "	12½	1.6 to 1.8
26	B552	" " "	14½	1.6 to 1.8
27	B326	" " "	11	0.45 to 0.55
28	B317	" " "	11	0.25 to 0.35
29	Vacant			
30	Vacant			

[a] Made from sample B443.

LIST II.—UNFABRICATED WIRES FOR TENSION TESTS (*Continued*).

FRAME No. 703, TENSION TESTS

Position of Wire on Frame	Sample Designation	Material	Gage (Steel Wire Gage)	Coating Classification
				Weight of Coating, oz. per sq. ft. of uncoated wire surface
1	B200	Non-copper-bearing steel	9	Bare
2	B346	Zinc-coated, copper-bearing steel	11	0.80 to 1.00
3	B347	" " "	11	0.80 to 1.00
4	B205	Copper-bearing steel	9	Bare
5	B206	" "	9	Bare
6	B215	Zinc-coated, copper-bearing steel	9	0.25 to 0.35
7	B216	" " "	9	0.25 to 0.35
8	B225	" " "	9	0.45 to 0.55
9	B227	" " "	9	0.45 to 0.55
10	B228	" " "	9	0.45 to 0.55
	Extra space between wires here			
11	B237	Zinc-coated, copper-bearing steel	9	0.60 to 0.70
12	B238	" " "	9	0.60 to 0.70
13	B239	" " "	9	0.60 to 0.70
14	B245	" " "	9	0.80 to 1.00
15	B246	" " "	9	0.80 to 1.00
16	B202	Non-copper-bearing steel	9	Bare
17	B345	Zinc-coated, copper-bearing steel	11	0.80 to 1.00
18	B348	" " "	11	0.80 to 1.00
19	B282	" " "	9	0.50 max.
20	B351	" " "	11	1.6 to 1.8
	Extra space between wires here			
21	B352	Zinc-coated, copper-bearing steel	11	1.6 to 1.8
22	B235	" " "	9	0.60 to 0.70
23	B236	" " "	9	0.60 to 0.70
24	B247	" " "	9	0.80 to 1.00
25	B248	" " "	9	0.80 to 1.00
26	B296[a]	" " "	9	0.80 to 1.00 chromated
27	B217	" " "	9	0.25 to 0.35
28	B350	" " "	11	1.6 to 1.8
29	B226	" " "	9	0.45 to 0.55
30	Vacant			

[a] Made from sample B245.

LIST II.—UNFABRICATED WIRES FOR TENSION TESTS (*Continued*).

FRAME NO. 704, TENSION TESTS

Position of Wire on Frame	Sample Designation	Material	Gage (Steel Wire Gage)	Coating Classification
				Weight of Coating, oz. per sq. ft. of uncoated wire surface
1	B101	Non-copper-bearing steel	6	Bare
2	B102	" "	6	Bare
3	B250	Zinc-coated, copper-bearing steel	9	1.6 to 1.8
4	B251	" " "	9	1.6 to 1.8
5	B105	Copper-bearing steel	6	Bare
6	B106	" "	6	Bare
7	B115	Zinc-coated, copper-bearing steel	6	0.25 to 0.35
8	B117	" " "	6	0.25 to 0.35
9	B125	" " "	6	0.45 to 0.55
10	B127	" " "	6	0.45 to 0.55
	Extra space between wires here			
11	B135	Zinc-coated, copper-bearing steel	6	0.60 to 0.70
12	B138	" " "	6	0.60 to 0.70
13	B139	" " "	6	0.60 to 0.70
14	B148	" " "	6	0.80 to 1.00
15	B149	" " "	6	0.80 to 1.00
16	B151	" " "	6	1.6 to 1.8
17	B299	" " "	9	2.4 min.
18	B800	" " "	6	1.2 to 1.5
19	B801	" " "	9	1.2 to 1.5
20	B252	" " "	9	1.6 to 1.8
	Extra space between wires here			
21	B298	Zinc-coated, copper-bearing steel	9	2.4 min.
22	B397	" " "	11	2.4 min.
23	Vacant			
24	Vacant			
25	Vacant			
26	Vacant			
27	Vacant			
28	Vacant			
29	Vacant			
30	Vacant			

LIST II.—UNFABRICATED WIRES FOR TENSION TESTS (*Continued*).

FRAME NO. 705, TENSION TESTS

Position of Wire on Frame	Sample Designation	Material	Gage (Steel Wire Gage)
1	B001	Corrosion-resistant steel, 12 to 14 per cent chromium, cold-drawn wire	12
2	Vacant		
3	B002	Corrosion-resistant steel, 12 to 14 per cent chromium, air-quenched wire	$12\frac{1}{2}$
4	Vacant		
5	B003	Corrosion-resistant steel, 18 per cent chromium, 8 per cent nickel	15
6	Vacant		
7	B004	Corrosion-resistant steel, 12 to 14 per cent chromium, cold-drawn wire	9
8	Vacant		
9	B005	Corrosion-resistant steel, 12 to 14 per cent chromium, cold-drawn wire	17
10	Vacant		
	Extra space between wires here		
11	B006	Corrosion-resistant steel, 12 to 14 per cent chromium, air-quenched wire	10
12	Vacant		
13	B007	Corrosion-resistant steel, 12 to 14 per cent chromium, air-quenched wire	18
14	Vacant		
15	B008	Corrosion-resistant steel, 18 per cent chromium, 8 per cent nickel	12
16	Vacant		
17	B009	Corrosion-resistant steel, 18 per cent chromium, 8 per cent nickel	20
18	Vacant		
19	B015	Corrosion-resistant steel, 18 per cent chromium, 8 per cent nickel	14
20	Vacant		
	Extra space between wires here		
21	B021	Copper-covered steel	11
22	B022	Copper-covered steel	9
23	Vacant		
24	B040	Lead-coated steel	6
25	B041	Lead-coated steel	9
26	B042	Lead-coated steel	11
27	B043	Lead-coated steel	$14\frac{1}{2}$
28	Vacant		
29	Vacant		
30	Vacant		

NOTE.—Shortage in Specimens of Unfabricated Wire:

At Lafayette, Ind.	No. 495 missing on Frame No. B702
At College Station, Tex.	No. 296 missing on Frame No. B703
At Davis, Calif.	No. 015 missing on Frame No. B705
	No. 296 missing on Frame No. B703
	No. 299 missing on Frame No. B704
	No. 495 missing on Frame No. B702
	No. 800 missing on Frame No. B704
	No. 801 missing on Frame No. B704
At Santa Cruz, Calif.	No. 015 missing on Frame No. B705
	No. 296 missing on Frame No. B703
	No. 299 missing on Frame No. B704
	No. 495 missing on Frame No. B702
	No. 800 missing on Frame No. B704
	No. 801 missing on Frame No. B704

LIST III.—UNFABRICATED WIRES FOR WEIGHT LOSS TESTS.

Position on Frame	Frames on East Side of Racks				Position on Frame	Frames on West Side of Racks			
	Sandy Hook					Sandy Hook			
	Frame No. BB710-01	Frame No. BB710-02	Frame No. BB710-03	Frame No. BB710-04		Frame No. BB711-01	Frame No. BB711-02	Frame No. BB711-03	Frame No. BB711-04
1	B148-01	B148-03	B148-05	B148-07	1	B148-09	B148-11	B148-13	B148-15
2	B148-02	B148-04	B148-06	B148-08	2	B148-10	B148-12	B148-14	B148-16
3	B346-01	B346-03	B346-05	B346-07	3	B346-09	B346-11	B346-13	B346-15
4	B346-02	B346-04	B346-06	B346-08	4	B346-10	B346-12	B346-14	B346-16
5	B542-01	B542-03	B542-05	B542-07	5	B542-09	B542-11	B542-13	B542-15
6	B542-02	B542-04	B542-06	B542-08	6	B542-10	B542-12	B542-14	B542-16
7	B247-01	B247-03	B247-05	B247-07	7	B247-09	B247-11	B247-13	B247-15
8	B247-02	B247-04	B247-06	B247-08	8	B247-10	B247-12	B247-14	B247-16
9	B345-01	B345-03	B345-05	B345-07	9	B345-09	B345-11	B345-13	B345-15
10	B345-02	B345-04	B345-06	B345-08	10	B345-10	B345-12	B345-14	B345-16
11	B541-01	B541-03	B541-05	B541-07	11	B541-09	B541-11	B541-13	B541-15
12	B541-02	B541-04	B541-06	B541-08	12	B541-10	B541-12	B541-14	B541-16
13	B248-01	B248-03	B248-05	B248-07	13	B248-09	B248-11	B248-13	B248-15
14	B248-02	B248-04	B248-06	B248-08	14	B248-10	B248-12	B248-14	B248-16
15	B348-01	B348-03	B348-05	B348-07	15	B348-09	B348-11	B348-13	B348-15
16	B348-02	B348-04	B348-06	B348-08	16	B348-10	B348-12	B348-14	B348-16
17	B540-01	B540-03	B540-05	B540-07	17	B540-09	B540-11	B540-13	B540-15
18	B540-02	B540-04	B540-06	B540-08	18	B540-10	B540-12	B540-14	B540-16

Position on Frame	Pennsylvania State College				Position on Frame	Pennsylvania State College			
	Frame No. DB710-05	Frame No. DB710-06	Frame No. DB710-07	Frame No. DB710-08		Frame No. DB711-05	Frame No. DB711-06	Frame No. DB711-07	Frame No. DB711-08
1	B148-17	B148-19	B148-21	B148-23	1	B148-25	B148-27	B148-29	B148-31
2	B148-18	B148-20	B148-22	B148-24	2	B148-26	B148-28	B148-30	B148-32
3	B346-17	B346-19	B346-21	B346-23	3	B346-25	B346-27	B346-29	B346-31
4	B346-18	B346-20	B346-22	B346-24	4	B346-26	B346-28	B346-30	B346-32
5	B542-17	B542-19	B542-21	B542-23	5	B542-25	B542-27	B542-29	B542-31
6	B542-18	B542-20	B542-22	B542-24	6	B542-26	B542-28	B542-30	B542-32
7	B247-17	B247-19	B247-21	B247-23	7	B247-25	B247-27	B247-29	B247-31
8	B247-18	B247-20	B247-22	B247-24	8	B247-26	B247-28	B247-30	B247-32
9	B345-17	B345-19	B345-21	B345-23	9	B345-25	B345-27	B345-29	B345-31
10	B345-18	B345-20	B345-22	B345-24	10	B345-26	B345-28	B345-30	B345-32
11	B541-17	B541-19	B541-21	B541-23	11	B541-25	B541-27	B541-29	B541-31
12	B541-18	B541-20	B541-22	B541-24	12	B541-26	B541-28	B541-30	B541-32
13	B248-17	B248-19	B248-21	B248-23	13	B248-25	B248-27	B248-29	B248-31
14	B248-18	B248-20	B248-22	B248-24	14	B248-26	B248-28	B248-30	B248-32
15	B348-17	B348-19	B348-21	B348-23	15	B348-25	B348-27	B348-29	B348-31
16	B348-18	B348-20	B348-22	B348-24	16	B348-26	B348-28	B348-30	B348-32
17	B540-17	B540-19	B540-21	B540-23	17	B540-25	B540-27	B540-29	B540-31
18	B540-18	B540-20	B540-22	B540-24	18	B540-26	B540-28	B540-30	B540-32

Position on Frame	Pittsburgh				Position on Frame	Pittsburgh			
	Frame No. EB710-09	Frame No. EB710-10	Frame No. EB710-11	Frame No. EB710-12		Frame No. EB711-09	Frame No. EB711-10	Frame No. EB711-11	Frame No. EB711-12
1	B148-33	B148-35	B148-37	B148-39	1	B148-41	B148-43	B148-45	B148-47
2	B148-34	B148-36	B148-38	B148-40	2	B148-42	B148-44	B148-46	B148-48
3	B346-33	B346-35	B346-37	B346-39	3	B346-41	B346-43	B346-45	B346-47
4	B346-34	B346-36	B346-38	B346-40	4	B346-42	B346-44	B346-46	B346-48
5	B542-33	B542-35	B542-37	B542-39	5	B542-41	B542-43	B542-45	B542-47
6	B542-34	B542-36	B542-38	B542-40	6	B542-42	B542-44	B542-46	B542-48
7	B247-33	B247-35	B247-37	B247-39	7	B247-41	B247-43	B247-45	B247-47
8	B247-34	B247-36	B247-38	B247-40	8	B247-42	B247-44	B247-46	B247-48
9	B345-33	B345-35	B345-37	B345-39	9	B345-41	B345-43	B345-45	B345-47
10	B345-34	B345-36	B345-38	B345-40	10	B345-42	B345-44	B345-46	B345-48
11	B541-33	B541-35	B541-37	B541-39	11	B541-41	B541-43	B541-45	B541-47
12	B541-34	B541-36	B541-38	B541-40	12	B541-42	B541-44	B541-46	B541-48
13	B248-33	B248-35	B248-37	B248-39	13	B248-41	B248-43	B248-45	B248-47
14	B248-34	B248-36	B248-38	B248-40	14	B248-42	B248-44	B248-46	B248-48
15	B348-33	B348-35	B348-37	B348-39	15	B348-41	B348-43	B348-45	B348-47
16	B348-34	B348-36	B348-38	B348-40	16	B348-42	B348-44	B348-46	B348-48
17	B540-33	B540-35	B540-37	B540-39	17	B540-41	B540-43	B540-45	B540-47
18	B540-34	B540-36	B540-38	B540-40	18	B540-42	B540-44	B540-46	B540-48

LIST IV.—BARBED WIRE.[a]

Fence Number	Sample Designation	Material	Coating Classification
			Weight of Coating, oz. per sq. ft. of uncoated wire surface
2	C405	Copper-bearing steel	Bare
	C406	" "	Bare
	C410	Zinc-coated, copper-bearing steel	0.20 to 0.30
	C411	" " "	0.20 to 0.30
3	C412	" " "	0.20 to 0.30
	C421	" " "	0.35 to 0.45
	C422	" " "	0.35 to 0.45
	C420	" " "	0.35 to 0.45
	C430	" " "	0.50 to 0.60
	C431	" " "	0.50 to 0.60
	C432	" " "	0.50 to 0.60
	C441	" " "	0.70 to 0.85
4	C442	" " "	0.70 to 0.85
	C443	" " "	0.70 to 0.85
5	C445	" " "	0.85 to 1.00
	C446	" " "	0.85 to 1.00
	C447	" " "	0.85 to 1.00
6	C450	" " "	1.6 min.
	C452	" " "	1.6 min.
	C451	" " "	1.6 min.
8	C494	" " "	2.4 to 2.6
	C025	Copper-covered steel	......
	C045	Lead-coated steel	......

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[a] There is only one length each of samples C432 and C451 at College Station, Tex.

LIST V.—WIRE STRAND.

Sample Designation	Material	Coating Classification
		Weight of Coating, oz. per sq. ft. of uncoated wire surface
D345	Zinc-coated, Siemens-Martin steel	0.80 to 0.95
D346	" " " "	0.80 to 0.95
D347	" " " "	0.80 to 0.95
D348	" " " "	0.80 to 0.95
D360	" " " "	1.0 to 1.2
D361	" " " "	1.0 to 1.2
D362	" " " "	1.0 to 1.2
D365	" " " "	1.3 to 1.5
D366	" " " "	1.3 to 1.5
D367	" " " "	1.3 to 1.5
D371	" " " "	1.6 to 1.8
D375	" " " "	2.4 to 2.6
D045	Lead-coated steel	......

LIST VI.—CHAIN-LINK FENCE.

Fence Number[a]	Sample Designation	Material	Galvanized Before or After Weaving	Gage (Steel Wire Gage)	Coating Classification
					Weight of Coating, oz. per sq. ft. of uncoated wire surface
9a	E280	Zinc-coated, copper-bearing steel	Before	9	0.50 max.
9a	E281	" " "	Before	9	0.50 max.
9a	E282	" " "	Before	9	0.50 max.
9a	H280	" " "	After	9	0.50 max.
9b	E185	Zinc-coated, copper-bearing steel	Before	6	1.20 to 1.50
9b	H185	" " "	After	6	1.20 to 1.50
9b	H186	" " "	After	6	1.20 to 1.50
9b	H187	" " "	After	6	1.20 to 1.50
9b	H188	" " "	After	6	1.20 to 1.50
9b	E290	" " "	Before	9	2.4 to 3.0
9b	E291	" " "	Before	9	2.4 to 3.0
9b	H290	" " "	After	9	2.4 to 3.0
10a	E285	Zinc-coated, copper-bearing steel	Before	9	1.2 to 1.5
10a	H285	" " "	After	9	1.2 to 1.5
10a	H286	" " "	After	9	1.2 to 1.5
10a	H287	" " "	After	9	1.2 to 1.5
10a	H288	" " "	After	9	1.2 to 1.5
10a	H289	" " "	After	9	1.2 to 1.5
10a	L050	Lead-coated steel	....	9	....
10b	J011	Corrosion-resistant steel	....	14	....
10b	K030	Copper-covered steel	....	0.128 in. diameter	....

[a] The two sections of Fence 9 and Fence 10, respectively, are separated by braced "end" posts.

REPORT OF COMMITTEE A-7
ON
MALLEABLE IRON CASTINGS

Committee A-7 on Malleable Iron Castings has held one meeting during the year, on June 29, 1936, at Atlantic City, N. J., during the annual meeting of the Society. At this meeting a set of proposed by-laws was presented and adopted. An election of officers for the ensuing term of two years resulted in the selection of E. K. Smith, chairman; H. W. Faus, vice-chairman; and H. A. Schwartz, secretary. During the year Mr. Faus resigned as a member of the committee and the vacancy in the vice-chairmanship has not yet been filled.

It is with regret that the committee records the death of Mr. J. C. Haswell, President of the Dayton Malleable Iron Co., and of Mr. J. C. Ramage, Engineer of Tests of the Southern Railway Co., both of whom were long-time members of the committee.

Three members of the committee have resigned and ten have been elected to membership, which brings the total personnel of the committee to 38 members, of whom 17 are classified as producers, 13 as consumers, and 8 as general interest members.

Subsequent to the 1936 annual meeting, Committee A-7 presented to the Society through Committee E-10 on Standards, proposed Tentative Specifications for Cupola Malleable Iron (A 197 – 36 T) which had been prepared by Subcommittee III on Cupola Malleable Iron (R. E. Bryant, chairman). The proposed tentative specifications were accepted[1] by Committee E-10 on August 26, 1936, and appear in the 1936 *Proceedings*.[2]

Subcommittee I on Nomenclature and Definitions (H. A. Schwartz, chairman) has approved the definitions of "malleable cast iron," and "pearlitic malleable cast iron" which appear in the Tentative Definitions of Terms Relating to Cast Iron (A 196 – 36 T),[3] prepared by Committee A-3 on Cast Iron.

At the meeting of the committee held on June 29, 1936, an interesting report on galvanizing embrittlement was presented by Mr. Enrique Touceda, who at that time was chairman of Subcommittee II on Galvanizing

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[1] In submitting these tentative specifications to Committee E-10 on Standards, Committee A-7 reported results of the letter ballot vote as follows: Of a total membership of 33, 31 members returned their ballots, of whom 29 voted affirmatively, 1 negatively, and 1 member marked his ballot "not voting."

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 665 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 221.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 667 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 236.

Embrittlement. Subcommittee VI on Pearlitic and Alloy Malleable Iron (H. M. Boylston, chairman) has under consideration the preparation of proposed specifications for pearlitic malleable iron. These proposed specifications are to be discussed at the next meeting of Committee A-7 which is to be held in New York City during the annual meeting of the Society.

This report has been submitted to letter ballot of the committee which consists of 37 members; 35 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

E. K. SMITH,
Chairman.

H. A. SCHWARTZ,
Secretary.

REPORT OF COMMITTEE A-10

ON

IRON-CHROMIUM, IRON-CHROMIUM-NICKEL AND RELATED ALLOYS

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys has held one meeting during the year, in Chicago, Ill., on March 2, 1937, during the spring group meetings of A.S.T.M. committees. Several changes in committee personnel have occurred resulting in a present membership of 69, of whom 28 are classed as producers, 29 as consumers and 12 as general interest members.

Mr. C. E. MacQuigg resigned the vice-chairmanship of the committee and also the chairmanships of Subcommittee I on Classification of Data and of Subcommittee VI on Metallography. The selection of successors to Mr. MacQuigg as vice-chairman and the chairmen of these subcommittees is still to be made.

Because of interrelation of certain phases of work between Committee A-1 on Steel, Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys and Committee A-10, it became necessary to hold a joint conference to formulate statements of scope of the respective committees. These were drawn up for submission to the Executive Committee of the Society, the following being outlined for Committee A-10:

> *Scope.*—In general, Committee A-10 shall have jurisdiction over the classification, specification requirements, methods of test, questions of utility and related subjects pertaining to those iron-base alloys termed "stainless" or "rustless" whose primary characteristic is resistance to corrosive attack, elevated temperatures or both.
>
> The scopes of Committees A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys, B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys, and A-1 on Steel cannot be definitely limited. Therefore, a coordinating committee consisting of representatives of each of these committees will decide questions of jurisdiction over specific materials and applications, subject to the approval of the committees concerned.

The by-laws governing Committee A-10 have been found inadequate to provide for selection of officers following resignations, and also in other respects, and will be reviewed and amended by a committee appointed by the chairman.

Subsequent to the 1936 annual meeting, Committee A-10 presented to the Society through Committee E-10 on Standards proposed Tentative Specifications for 20 per cent Chromium, 9 per cent Nickel Alloy-Steel

Castings (A 198 – 36 T). These specifications were accepted[1] for publication by Committee E-10 on October 14, 1936, and appear in the 1936 *Proceedings*.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Classification of Data (C. E. MacQuigg, ex-chairman).—This subcommittee was reorganized during a recent meeting and Russell Franks was elected secretary. The classification of data will be carried out by subgroups under a chairman for each group. The personnel of each of the subgroups who will collect and correlate the data for publication will consist of a group of authorites and individuals having expert knowledge on the particular alloys. Tentative arrangement of the groups, with chairmen, is as follows:

GROUP	ALLOY STEEL	CHAIRMAN
I.	5 to 10 per cent chromium	E. C. Wright
II.	11 to 16 per cent chromium	T. F. Olt
III.	16 to 20 per cent chromium	B. H. DeLong
IV.	20 to 35 per cent chromium	W. F. Furman
V.	18 per cent chromium, 8 per cent nickel	F. L. LaQue
VI.	24 per cent chromium, 12 per cent nickel[a]	R. J. Wilcox
VII.	20 per cent nickel, 9 per cent chromium[b]	Frank Garratt
VIII.	35 per cent nickel, 15 per cent chromium	W. J. Jeffries

[a] Or other ratios with chromium predominating (and excluding Group V).
[b] Or other ratios with nickel predominating.

The final form for charting the data has been agreed upon and immediate activity in preparation of the data can be expected.

Subcommittee IV on Methods of Corrosion Testing (E. S. Taylerson, chairman).—Further discussion of this subcommittee's program at a meeting in Chicago, Ill., seems generally to indicate that exposure tests on a small scale are favored. A program was presented for the initiation of a series of exposure tests to correlate composition, manufacturing and fabricating methods and exposure conditions. This is so broad that a special committee was appointed to study it and to report to the main committee on the part that Subcommittee IV can take in promoting this objective. A small subcommittee was outlined and is in process of formation to inspect existing installations and to correlate service with previous manufacturing history. Progress on both projects is expected by the time of the June meeting.

Other groups of the subcommittee will prepare recommended procedures for corrosion testing and compile information on the subject in an effort to standardize or rationalize such procedures.

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[1] In submitting these specifications to Committee E-10 on Standards, Committee A-10 reported results of the letter ballot vote as follows: Of a total membership of 63, 40 members returned their ballots, of whom 33 voted affirmatively, 2 negatively, and 5 marked their ballots "not voting."

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 639 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 133.

Subcommittee V on Mechanical Testing (V. N. Krivobok, chairman).—This subcommittee is continuing its program on the study of elastic properties of the 18 per cent chromium, 8 per cent nickel types of steel after various heat treatments.

Subcommittee VI on Metallography (C. E. MacQuigg, ex-chairman).—The work of this subcommittee is progressing and nearly all returns are in from a preliminary microscopic examination of samples of 18 per cent chromium, 8 per cent nickel steel after various heat treatments. Two heats containing 0.065 per cent and 0.098 per cent carbon are being examined and photomicrographs prepared at magnifications of 100 and 750 diameters. When this preliminary examination is completed, the subcommittee will continue its work in an effort to determine the nature of the grain boundary precipitation in austenitic steels of this type.

Subcommittee VII on Welding (L. C. Bibber, chairman).—Attempts to prepare specifications for welding have been abandoned and instead this subcommittee expects to compile information relating to welding the alloys falling within the scope of Committee A-10. The controversial nature of the subject has hampered progress to a certain extent and some further discussion of activity will be necessary before the work can be actively continued.

Subcommittee VIII on Specifications for Wrought Products (F. B. Foley, chairman).—This subcommittee is working on specifications for some of the types of products falling within its jurisdiction.

Subcommittee IX on Specifications for Flat Products (C. C. Snyder, chairman).—The three tentative specifications covering corrosion-resisting sheet, strip and plate prepared by this subcommittee are being continued in this status. Attention is being given to a revision of the Tentative Specifications for Corrosion-Resisting Chromium-Nickel Steels (Sheet, Strip and Plate) (A 167 – 35 T) in respect to the yield point and tensile strength requirements, and to the inclusion of requirements for columbium-treated 18 per cent chromium, 8 per cent nickel steel in these specifications.

Subcommittee X on Specifications for Castings (W. J. Jeffries, chairman).—The group of nine specifications for alloy-steel castings prepared by this subcommittee are being continued as tentative without revision. In the meantime, comments and experience in their use are being collected and revisions on the basis of these comments and experience are to be considered during the coming year. Much of the criticism thus far received has been directed to the Tentative Specifications for 20 per cent Chromium, 9 per cent Nickel Alloy Steel Castings (A 198 – 36 T),[1] which were accepted by Committee E-10 on October 14, 1936.

Subcommittee XI on Specifications for Tubular Products (Newell Hamilton, chairman).—The personnel of this subcommittee is being selected following which subgroups will be appointed to formulate specifications for the various tubular products.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 639 (1936); also 1936 Book of A.S.T.M. Tentative Standards ,p. 133.

This report has been submitted to letter ballot of the committee which consists of 69 members; 52 members returned their ballots, of whom 50 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
Chairman.

H. D. NEWELL,
Secretary.

REPORT OF THE RESEARCH COMMITTEE
ON
FATIGUE OF METALS

During the past year the cooperative arrangement that has been established by the committee with *Metals and Alloys* has been continued, and abstracts of current information concerning the fatigue of metals have appeared in that journal.

The committee held a meeting in Chicago, Ill., on March 2, 1937. The main part of the meeting was devoted to a summary of the problems in the field of fatigue and especially interesting investigations in progress. The desirability of conducting comparative fatigue tests using hollow and solid rotating-beam specimens and of comparing the test data with results of axial-tension-compression tests were mentioned but results are not yet available. Tests by W. M. Wilson at the University of Illinois on riveted joints under axial tension and compression using a testing machine with a maximum range of from 200,000 lb. per sq. in. in tension to 200,000 lb. per sq. in. in compression failed to produce failure by shear in the rivets. Failure occurred from a spreading crack starting at a bolt hole, although the calculated shearing stress was far above the shearing endurance limit of the steel in the rivets. Some discussion took place whether this shearing was due to the friction of the rivet heads, or to some other cause.

Fatigue failures in the case of repeated highly localized compressive stress, under such conditions as exist between a car wheel and a rail, were also discussed. Such failures include the "shelling" of the tread of car wheels, "pitting" of gear teeth, and internal fissures in railroad rails. Discussion as to the place where such failure starts brought out a probable explanation that it originates as the result of a shearing stress slightly below the surface at some minute crack or other imperfection in the metal. It was pointed out that under such intense compression the surface layers in contact become work-hardened, and therefore more brittle, and that a crack might start somewhere in the transition region between the work-hardened material and the material not work-hardened. It is interesting to note that H. E. Smith has indicated that the effect of "shelling" of wheels does not occur in the second wear, that is, after the wheel has been remachined and put back in service.

Tests on sheet metals for airplane use were reported to be in progress at the National Bureau of Standards. Special emphasis is placed on corrosion and erosion effects.

The significance of the shape of the upper branch of the *S-N* diagram was discussed. This led to the suggestion of a need for some cooperative research work in which a very uniform lot of material should be secured and test specimens machined and tested by various laboratories. In such an investigation exact records of the machining and polishing procedure used in preparing the specimens should be kept.

The matter of "size effect" with respect to stress concentration was discussed and reports from several laboratories were considered. Here again, the results are not complete enough for conclusions to be drawn.

The question of nomenclature for fatigue testing, especially of nomenclature for range of stress, was considered and it was decided that at the present time no action should be taken by the committee recommending any nomenclature as a proposed standard. Two types of nomenclature are in rather general use in this country and in England. They are discussed in an Appendix to this report which also includes a diagram proposed by R. L. Templin that is applicable to fatigue tests of those metals which do not develop a definite endurance limit, and for which the endurance limit must be stated for a definite number of cycles.

This report has been submitted to letter ballot of the committee which consists of 17 members; 16 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. F. MOORE,
Chairman.

APPENDIX

NOMENCLATURE FOR VARIOUS RANGES IN STRESS IN FATIGUE

This discussion of nomenclature is published as information only, and it is not intended to recommend any of the nomenclature noted here as a proposed standard.

At the present time in English-speaking countries two types of nomenclature are in rather common use, as follows:

1. The ratio between (numerically) minimum stress and maximum stress in a cycle is denoted by r. This ratio, r, may be either positive or negative. To specify the range of stress, the range ratio with its sign is to be given, and the (numerical) maximum stress together with the statement whether it is tension, compression, positive shear, or negative shear.
2. In the second system of nomenclature any cycle of stress is thought of as made up of a mean (steady) stress, S_s, with a reversed (alternating) stress, S_a, superimposed on it. To specify range of stress it is necessary to specify the mean stress and to specify whether it is tension, compression, positive shear, or negative shear and to specify the alternating stress in the cycle.

Table I shows how this nomenclature is applied in nine typical stress cycles.

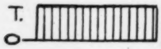
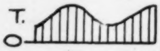
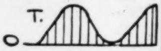
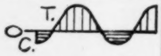
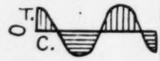
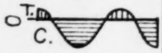
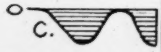
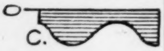
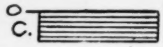
Numerous diagrams have been proposed representing the effect of range of stress. Perhaps the most common type would be the Goodman diagram slightly modified from its original form. Figure 1 shows such a diagram. The line CD represents the range for completely reversed stress. The stress in this cycle would be $\pm O'C$. AO represents a cycle in which the range ratio r is zero. $O''' F$ represents a cycle in which the ratio is $+\frac{O''' G}{O''' F}$. HK represents a cycle in which the maximum stress is $O''K$ and the range ratio r is $-\frac{O''H}{O''K}$.

Figure 2, which is self explanatory, shows a diagram based on the steady stress plus alternating stress nomenclature.

Figure 3 represents a diagram proposed by Earnest Hartman of the Aluminum Research Laboratories in which maximum stress in a cycle is plotted as ordinates, minimum stress as abscissas, and curves are drawn for endurance limits for various definite numbers of cycles. For example, for the given metal (a light alloy) under a cycle varying from 20,000 lb. per sq. in. in compression to 10,000 lb. per sq. in. in tension, the metal would be stressed below the endurance limit for 100,000,000 cycles of stress but above the limit for 500,000,000 cycles of stress. In this figure the terms "maximum" and "minimum" are used algebraically not numerically as in Paragraph 1 on the previous page.

TABLE I.—NUMERICAL EXAMPLES OF "RANGE RATIO" AND "MEAN STRESS" NOMENCLATURE FOR REPEATED STRESS.

In this table are given nine typical examples of the use of two systems of nomenclature for cycles of tension and compressive stress. A similar tabulation could be made for cycles of shearing stress, uni-directional and reversed. All values of stress are given in pounds per square inch and T. denotes tension, and C., compression.

Diagram of Stress During Cycle	Range of Stress Applied During Cycle	Range Ratio Nomenclature		Mean Stress Nomenclature	
		Range Ratio, r	Maximum Stress	Mean (steady) Stress, S_s	Reversed (alternating) Stress, S_a
	Steady stress, 30,000 T.	+1.0	30 000 T.	30 000 T.	0
	Pulsating stress from 10,000 T. to 25,000 T.	+0.4	25 000 T.	17 500 T.	±7 500
	Pulsating stress from 0 to 20,000 T.	0	20 000 T.	10 000 T.	±10 000
	Partly reversed stress from 5000 C. to 18,000 T.	−0.278	18 000 T.	6 500 T.	±11 500
	Completely reversed stress from 13,000 T. to 13,000 C.	−1.0	13 000 T. or C.	0	±13 000
	Partly reversed stress from 6000 T. to 22,000 C.	−0.273	22 000 C.	8 000 C.	±14 000
	Pulsating stress from 0 to 24,000 C.	0	24 000 C.	12 000 C.	±12 000
	Pulsating stress from 10,000 C. to 20,000 C.	+0.5	20 000 C.	15 000 C.	±5 000
	Steady stress, 35,000 C.	+1.0	35 000 C.	35 000 C.	0

There is a paucity of data available covering fatigue tests showing the effect of range of stress on shearing endurance limit. The results that are available indicate that the *range* of stress which will cause shearing fatigue failure is nearly independent of the maximum stress in a cycle. This is illustrated in Fig. 4 which is a modified Goodman diagram for shearing fatigue strength.

Experimental data for values of r approaching +1, or, what is the same thing, for values of alternating stress very small in comparison with the mean (steady) stress, are very few indeed. Various formulas have been proposed for expressing a ratio of endurance limit for different ranges of stress, but whether these formulas are the same for different metals is uncertain, nor is there much known about them for any metal for values of r between 0 and $+1$.

The study of the effect of range of stress is one of the most fertile fields in the study of fatigue of metals. It is time consuming, expensive and quite extensive. The ordinary rotating-beam test cannot readily be used, and the expensive direct axial tension-compression test is about the only test which will give reliable stress measurements when the metal is stressed into the plastic range.

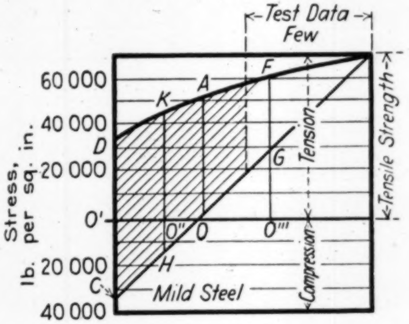


FIG. 1.—Modified Goodman Diagram.

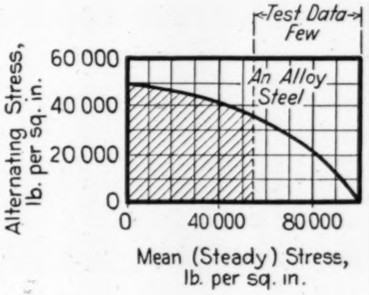


FIG. 2.—Mean Plus Alternating Stress Diagram (after Haigh).

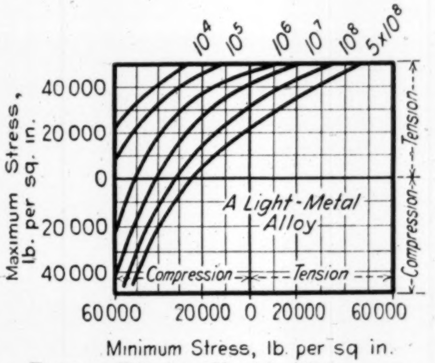


FIG. 3.—Diagram Used by Hartman.
Curves show endurance limits for different numbers of cycles of stress. In this figure the terms "maximum" and "minimum" are used in the algebraic sense; that is, a *negative* numerical maximum is an *algebraic* minimum.

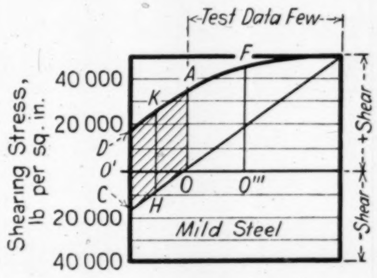


FIG. 4.—Modified Goodman Diagram for Shearing Stress (Torsion).

DISCUSSION

MR. R. E. PETERSON[1] (*presented in written form*).—It seems to the writer that the type of representation having mean stress as abscissas and maximum-minimum stress as ordinates (see accompanying Fig. 1) should also be mentioned. This type of diagram is somewhat similar to Fig. 2 of the report but has the advantage of showing the relationship of maximum and minimum stresses to the static properties of the material (proportional limit, yield strength, etc.). Incidentally, if the scales are the same, a 45-deg. line will be a line of symmetry and the combination of mean stress and alternating stress is clearly shown in the ordinate direction. This type of representation has been used to a considerable extent abroad, as for example in the V.D.I. design sheets, and it probably has a considerable usage in this country also. The writer has found, for example, that designers in the company with which he is associated like either this type of representation or the type of diagram indicated in Fig. 2 of the report.

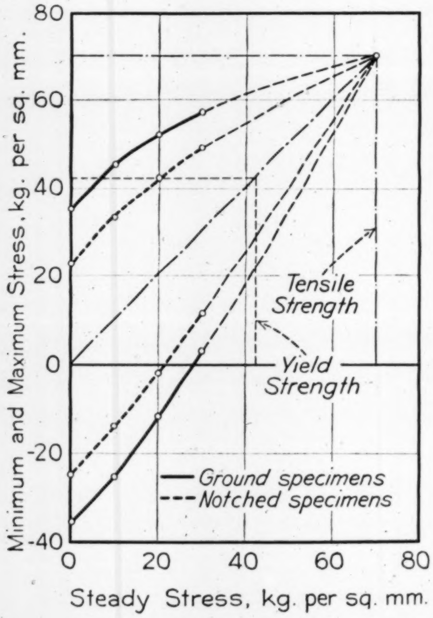


FIG. 1.—Notch Effect in Fatigue Stressing with Initial Tension (0.7 per cent Carbon Steel)
(According to Tests by Schenck, *Stahl und Eisen*, Vol. 52, p. 660)

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[1] Manager, Mechanics Division, Westinghouse Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

There is a further point which has a bearing on choice of representation. Such data as are available indicate that stress concentration factors apply to the alternating portion of the stress only (see accompanying Fig. 1). Various design codes, such as that by Soderberg,[1] are prepared on this principle. Method 2 of the report which really includes the type of representation shown in Fig. 2 of the report and also that illustrated by the accompanying Fig. 1, lends itself very well to the above consideration of stress concentration, while the other methods do not. It appears to me that this is not a minor point since nearly all members in practical use under variable stress conditions involve some form of stress concentration.

The writer wishes to emphasize that these are personal opinions only and he agrees with the viewpoint of not recommending any one procedure at this time. The real need is for more data from pulsating tests (at various mean stresses) of notched specimens. As far as the writer is aware, no work of this kind is now in progress, and it is hoped that someone will undertake such a program.

H. F. MOORE[2] (*closure by letter*).—I wish to thank Mr. Peterson both for the diagram he has presented and for the suggestion of its special usefulness in studying the relation of stress-concentration factor to range of stress during a cycle of repeated stress.

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[1] *Journal of Applied Mechanics*, September, 1935.

[2] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

REPORT OF JOINT RESEARCH COMMITTEE ON EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS

PROGRESS REPORT TO THE SPONSOR SOCIETIES

Meetings:

During the past year meetings were held on June 29 and 30 and July 1, 1936, in Atlantic City, N. J.; on November 30 and December 1, 1936, in New York City; on March 3, 1937, in Chicago, Ill.; and on June 4, 1937, in Columbus, Ohio.

Finances:

Beginning with the year 1932, a plan of sponsored researches was inaugurated and from then until the year 1935, funds were obtained amounting to about \$12,000[1] for the support of such work. Details of the researches and the contributions made were given in the annual reports[2] of 1932 to 1935, inclusive.

By the end of the year 1935, available funds had been expended and it became necessary to secure additional support for the committee's work. A new Finance Committee was appointed, under the chairmanship of R. A. Bull, and by June, 1936, had secured contributions of about \$10,000 from 22 industrial organizations and the Engineering Foundation, as was reported in detail in last year's annual report.[3] Additional funds were pledged during the past 12 months and now total \$19,500. Of these, payments totaling \$18,000 have been received, leaving a balance due of \$1500 (covering partial payments not yet due, on one subscription made for liquidation over a two-year period).

The contributors, of cash and the amounts pledged, are summarized below in the order in which the pledges were received by the committee:

1. The International Nickel Co., Inc.	\$1000
2. Ajax Electrothermic Corp.	500
3. Henry Vogt Machine Co., Inc.	200
4. Dodge Steel Co.	200
5. The Wm. Powell Co.	100

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[1] All moneys are deposited directly with the American Society of Mechanical Engineers which administers the fund under requisitions authorized by the Joint Committee.

[2] *Proceedings*, Am. Soc. Testing Mats., Vols. 32 to 35, Part I (1932 to 1935).

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 127 (1936).

6. Electro Metallurgical Co	$1000
7. Crane Co	500
8. Lebanon Steel Foundry	200
9. Bethlehem Steel Co., Inc	750
10. Stockham Pipe Fittings Co	200
11. Sivyer Steel Casting Co	200
12. Climax Molybdenum Co	750
13. Standard Oil Development Co	500
14. American Chain Co., Inc	350
15. The Texas Company	500
16. The Midvale Co	250
17. General Electric Co	1000
18. Allis-Chalmers Manufacturing Co	250
19. The Edward Valve and Manufacturing Co., Inc	100
20. Westinghouse Electric and Manufacturing Co	750
21. The Lunkenheimer Co	250
22. The Engineering Foundation	500
23. The Standard Oil Co. (Ohio)	100
24. The Babcock & Wilcox Co	400
25. Allegheny Steel Co	400
26. E. I. du Pont de Nemours and Co., Inc	500
27. Standard Oil Company of California	250
28. The Superheater Co	250
29. United States Steel Corp	750
30. The Carpenter Steel Co	100
31. Utilities Coordinated Research, Inc	3000
32. Republic Steel Corp	500
33. Walworth Co., Inc	300
34. The Calorizing Co	200
35. American Petroleum Institute[a]	2700
Total	$19,500

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[a] The American Petroleum Institute was requested to support the work of the committee subsequent to the receipt of contributions from some individual oil companies. The Institute agreed to solicit funds from the industry, and the contribution of $2700 represents the contributions obtained from companies other than those listed above.

Expenditures since June, 1936, included: $2250 at Battelle Memorial Institute for project No. 12;[1] $500 granted by Engineering Foundation to initiate project No. 18; $1500 at the University of Michigan for project No. 11; $1000 at the Massachusetts Institute of Technology to initiate project No. 10.

A detailed statement of the finances of the committee is given in the following balance sheet which shows $14,447 available for the Joint Committee's program.

CASH RECEIPTS (SINCE MAY 31, 1936)

Subscriptions	$12 000.00
Interest	76.06
Total	$12 076.06

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[1] For a key to the nature of the different projects, see later sections of this report.

EXPENDITURES (SINCE MAY 31, 1936)

1936		
June	None	
July	Postage (A.S.M.E. Office)	$ .22
	Battelle Memorial Institute (Project No. 12)	1000.00
	Finance Committee (Expenses)	200.00
	University of Michigan (Project No. 11)	500.00
August	Postage (A.S.M.E. Office)	.70
September	University of Michigan (Project No. 11)	500.00
	Ogilvie Press (Printing)	8.93
October	Battelle Memorial Institute (Project No. 12)	500.00
November	Postage (A.S.M.E. Office)	.22
December	Finance Committee (Expenses)	63.52
1937		
January	Messenger Service (A.S.M.E. Office)	.70
	Battelle Memorial Institute (Project No. 12)	500.00
	Massachusetts Institute of Technology (Project No. 10)	1000.00
February	Battelle Memorial Institute (Project No. 17)	24.31
March	University of Michigan (Project No. 11)	500.00
	Battelle Memorial Institute (Project No. 12)	250.00
April	Battelle Memorial Institute (Project No. 18)[a]	500.00
		$5548.38

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[a] A direct payment of $500 was made from Engineering Foundation to Battelle Memorial Institute (on project No. 18). This is shown in the above balance sheet as both a receipt and an expenditure.

Balance on hand May 31, 1936	$7 919.42
Cash receipts since May 31, 1936	12 076.06
	$19 995.48
Expenditures since May 31, 1936	5 548.38
Balance on hand	$14 447.10

Contributions of Materials:

Contributions of materials for use in the various projects of the committee have been made as follows:

Crucible Steel Company of America	Approximately 3500 lb. of 4 to 6 per cent chromium, 0.5 per cent molybdenum steel. Approximately 4200 lb. of carbon-molybdenum steel. Both in bar form.
Babcock & Wilcox Tube Co.	Processing the above two materials into tubular form.

Organization:

The funds available permitted appreciable enlargement of the program of sponsored researches and resulted in a heavy burden being placed on a few of the members of the committee under the plan of organization adopted in 1932. Reorganization was believed to be desirable, so as to permit a more equitable distribution of responsibility and work among the membership and this was undertaken during the year.

It was decided that there should be no standing committees within the Joint Committee, other than the Executive Committee, but as projects were authorized a chairman of a "Special Committee" would be appointed to handle the project in question, including the formation of the Special Committee. Upon completion of a project, the Special Committee would be automatically discharged.

This plan of organization was put into effect during March, 1937, and, with the exception of the Finance Committee and the reappointment of committee chairmen handling individual projects, all committees and subcommittees previously appointed were discharged.

The officers of the committee now comprise a Chairman, a Vice-Chairman, and a Secretary. N. L. Mochel, who since 1929 has been Secretary of the Joint Committee, was elected Vice-Chairman; J. W. Bolton was made Secretary.

The Executive Committee is comprised of the three officers of the Joint Committee and two additional members. The two members-at-large, elected in March, 1937, were F. B. Foley and E. L. Robinson.

Election of officers and two members of the Executive Committee is to take place every two years, in the even numbered years.

Membership of the Joint Committee, as of June, 1937, comprises the following:

R. H. Aborn, Research Laboratory, U. S. Steel Corp., Kearny, N. J.
W. H. Armacost, Combustion Engineering Co., Inc., 200 Madison Ave., New York City.
A. B. Bagsar, Chief Metallurgist, Sun Oil Co., Marcus Hook, Pa.
A. D. Bailey, Chief Operating Engineer, Commonwealth Edison Co., 72 W. Adams St., Chicago, Ill.
F. E. Bash, Manager, Technical Dept., Driver-Harris Co., Harrison, N. J.
J. W. Bolton, Chief Chemist and Metallurgist, The Lunkenheimer Co., Cincinnati, Ohio.
R. A. Bull, Consultant on Steel Castings, 541 Diversey Parkway, Chicago, Ill.
The Director, National Bureau of Standards, Department of Commerce, Washington, D. C.
The Director, Naval Engineering Experiment Station, Annapolis, Md.
E. S. Dixon, Metallurgist, The Texas Company, Port Arthur, Texas.
F. B. Foley, Director of Research, The Midvale Co., Nicetown, Philadelphia, Pa.
J. R. Freeman, Jr., Assistant Technical Manager, American Brass Co., Waterbury, Conn.
H. J. French, In Charge, Alloy Steel and Iron Development, The International Nickel Co., Inc., 67 Wall St., New York City.
H. W. Gillett, Chief Technical Adviser, Battelle Memorial Institute, Columbus, Ohio.
A. J. Herzig, Chief Metallurgist, Climax Molybdenum Co., Detroit, Mich.
G. F. Jenks, Colonel, Office of Chief of Ordnance, U. S. Army, Munitions Building, Washington, D. C.
J. J. Kanter, Crane Co., 836 S. Michigan Ave., Chicago, Ill.
H. J. Kerr, The Babcock & Wilcox Co., Barberton, Ohio.
C. E. MacQuigg, Union Carbide and Carbon Research Laboratories, 30 E. 42nd St., New York City.
P. E. McKinney, Metallurgical Engineer, Bethlehem Steel Co., Bethlehem, Pa.
N. L. Mochel, Metallurgical Engineer, Westinghouse Electric and Manufacturing Co., Lester Branch P. O., Philadelphia, Pa.
E. L. Robinson, Turbine Dept., General Electric Co., Schenectady, N. Y.
A. E. White, Director, Department of Engineering Research, University of Michigan, Ann Arbor, Mich.

There follows a list of the members of the committees handling the different portions of the Joint Committee's work:

Executive Committee (to June, 1938)
- H. J. French
- N. L. Mochel
- J. W. Bolton
- F. B. Foley
- E. L. Robinson

Committee on Finances (to June, 1938)
- R. A. Bull (Chairman)
- A. D. Bailey
- E. S. Dixon
- H. J. French
- C. E. MacQuigg
- E. L. Robinson

Project No. 10—Testing of Tubular Members at Massachusetts Institute of Technology
- C. E. MacQuigg (Chairman)
- F. H. Norton

Project No. 11—Assembly and Interpretation of Available Creep Data
- P. E. McKinney (Chairman)
- F. E. Bash
- C. L. Clark
- J. J. Kanter
- H. J. Kerr
- N. L. Mochel
- E. L. Robinson

Project No. 12—Long-Time Creep Tests on K20 Steel at Battelle Memorial Institute
- H. W. Gillett (Chairman)

Project No. 13—Properties of Metals at Low Temperature
- H. W. Russell (Chairman)

Project No. 14—Revision of Tentative Methods E 22 - 35 T
- C. L. Clark (Chairman)
- F. B. Foley (Secretary)
- R. H. Aborn
- A. C. Chamberlin
- L. W. Clark
- H. C. Cross
- J. J. Kanter
- M. S. Northrup
- E. L. Robinson
- E. L. Roff
- J. B. Romer and C. O. Burgess
- C. A. Scharschu
- David Zuege

Project No. 15—Torsion-Creep Tests at University of Michigan
- A. E. White (Chairman)
- F. L. Everett

Project No. 16—Relaxation Tests
- E. L. Robinson (Chairman)
- J. J. Kanter
- N. L. Mochel

Project No. 17—Life Test Methods
- A. E. White (Chairman)

Project No. 18—Effect of Manufacturing Variables
- H. W. Gillett (Chairman)

Project No. 19—Procurement of Material for Projects Nos. 10, 15, 16 and 17
- H. J. Kerr (Chairman)
- R. A. Bull

High-Temperature Test Methods:

The Tentative Methods of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 - 34 T) and for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 - 35 T) were reviewed during the year.

The subcommittee formerly known as III-E has reviewed Method E 22 and has initiated further research work among cooperating laboratories before recommending revisions of the method. The further consideration of this method has now been listed as project No. 14, and the chairman and members of former Subcommittee III-E have been appointed as the committee for project No. 14. The Joint Committee accordingly recommends that Method E 22 be continued as tentative.

The subcommittee formerly known as III-D, under the chairmanship of H. J. Kerr, completed its review of Method E 21 and has submitted a number of recommended changes in the method, as given in detail in Appendix I to this report. A.S.T.M. Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys has prepared an addendum to Method E 21, as given in Appendix II to this report, relating to the preparation of cast tension test specimens of heat-resistant metals (chromium-nickel and chromium-nickel-iron alloys) to be tested in accordance with this method. The Joint Committee accordingly recommends that the Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 34 T) be revised in accordance with the detailed changes given in Appendices I and II, and continued as tentative.

This recommendation has been submitted to letter ballot of the Joint Committee which consists of 22 members, for submission to the sponsor societies; all members returned their ballots, with the following results:

Item	Affirmative	Negative	Ballots Marked "Not Voting"
PROPOSED REVISION OF TENTATIVE STANDARD			
Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 34 T):			
Revisions of tension test as given in Appendix I	19	0	3
Inclusion of addendum on preparation of cast tension test specimens of heat-resistant metals as given in Appendix II	19	0	3

Research Projects:

There follows a summary of the various new research projects and of work previously initiated and continued during 1936–1937.

Project No. 10 on Testing of Tubular Members at Elevated Temperatures at the Massachusetts Institute of Technology (C. E. MacQuigg, chairman).—This work is to permit comparisons of the creep of steels at elevated temperatures under biaxial stresses (tube tests) and under unidirectional stress (usual tension creep tests). Biaxial stresses will be obtained by internal pressure in tubes in special testing equipment now under construction at the Massachusetts Institute of Technology, under the supervision of F. H. Norton.

The preliminary tests now authorized are on carbon-molybdenum steel, at two loads at each of two temperatures, 800 and 1050 F.; also on 5 per cent chromium steel, containing molybdenum, to be tested at two loads at 1200 F.

Expenditures during a period of two years were authorized up to $6000.

Project No. 11 on Assembly and Interpretation of Available Creep Test Data at the University of Michigan (P. E. McKinney, chairman).—The selection of metals for equipment operating at elevated temperatures often requires a knowledge of their creep characteristics. Technical literature reveals large differences in the creep of steels not readily explainable by the differences in testing procedure or by variations in metal composition.

A compilation, under C. L. Clark, at the University of Michigan, of published and unpublished creep test data was authorized so as to secure for engineers a better idea of the creep characteristics of commonly used metals. Requests for unpublished data were directed to some 45 American and 22 foreign laboratories and when received the information will be coordinated with published data already assembled. Subsequently, an analysis of the data will be made.

To date, extensive data including over 300 creep tests have been contributed by American Brass Co., The Babcock & Wilcox Tube Co., Battelle Memorial Institute, Bethlehem Steel Co., Brown-Boveri and Co., Ltd., Climax Molybdenum Co., Crane Co., General Electric Co., Jacob Holtzer, The International Nickel Co., Inc., The Lunkenheimer Co., National Tube Co., Timken Steel & Tube Co., University of Illinois, National Physical Laboratory (England), and The Technische Hochschule, Breslau, Germany.

Expenditures to date total $1500 and it is contemplated that the work will be completed by January 1, 1938, at a cost not in excess of $500 additional.

Project No. 12 on Long-Time Creep Tests on Carbon Steel (*K20*) *at Battelle Memorial Institute* (H. W. Gillett, chairman).—This project, which has been under way for several years, is concerned with the validity of tests of several hundred and several thousand hours' duration as indicators of creep "strength" for practical service life of 20,000 hr. or more.

Tests have been under way on a carbon steel (K20) at 850 F. and an austenitic chromium-nickel steel (K19) at 1150 F. Work on the latter was discontinued during the year because the total creep exceeded the amounts in which many users are interested. The test on one of the carbon-steel bars (K20) is still under way, the load and temperature having been maintained continuously for more than 17,000 hr.

A detailed progress report of this research, by H. C. Cross and J. G. Lowther, comprises Appendix III to this report, entitled "Report on Long-Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel and 0.35 per cent Carbon Steel."

Project No. 13 on Investigations of the Properties of Metals at Low Temperatures (H. W. Russell, chairman).—The work of this group, so far, has been restricted to the preparation of a questionnaire and an analysis of the replies intended to bring out problems of greatest general interest. Specific researches have not yet been planned and no appropriations or expenditures have been made.

Project No. 14 on Revision of Tentative Method E 22 – 35 T (C. L. Clark, chairman).—Further consideration will be given during the year to the Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 – 35 T), as mentioned earlier in this report.

Project No. 15 on Torsion-Creep Tests for Comparison with Tension-Creep Tests (A. E. White, chairman).—Expenditures not in excess of $1200 were authorized for work which will be started in the near future, under F. L. Everett, at the University of Michigan. Tests will be made on carbon-molybdenum steel at four loads at each of two temperatures, 800 and 1050 F.

It is hoped that the torsion-creep tests may throw further light upon plastic flow phenomena and that possibly the results may be of a nature which will assist in the analysis of creep under compound stress.

Project No. 16 on Relaxation Tests (E. L. Robinson, chairman).—In connection with the work under projects Nos. 10 and 15, relaxation tests are planned on samples of the carbon-molybdenum steel for comparison with the torsion-creep tests, also tension-creep tests made under the usual conditions. Relaxation tests also will be made on chromium-molybdenum steel bolting stock (S.A.E. No. 4140).

Aside from the correlation of relaxation test data with the results of other tests, the data are expected to be of direct interest to engineers because they will represent different conditions of stress and temperature encountered in practice. Expenditures up to $800 have been authorized.

Project No. 17 on Study of High-Temperature Life-Test Methods (A. E. White, chairman).—Expenditures up to $1000 have been authorized for work under C. L. Clark at the University of Michigan. The time for fracture of carbon steel (K20) and carbon-molybdenum steel will be determined under conditions of relatively rapid flow (about 24 hr. or less for fracture), with the idea that definite and determinable relations exist between stress and temperature which may be utilized as the basis for life tests of metals and which may, in a short period of time, give a reasonably good indication of long-time creep "strength." The results will be coordinated with constant strain-rate tests on the carbon-molybdenum steel made by A. Nadai, through the courtesy of Westinghouse Electric and Manufacturing Co.

Samples of the carbon steel (K20) have also been forwarded to the University of Brussels, where modified Pomp and Dahmen tests will be made, the results to be correlated with the information referred to above, so as to secure a broader picture of the several accelerated test methods proposed during recent years for rapid determinations of creep properties of steels at elevated temperatures.

Project No. 18 on Study of Effects of Manufacturing Variables on the Creep Resistance of Steels (H. W. Gillett, chairman).—This problem, which seems to many of the members of the Joint Committee to be of fundamental importance, is discussed in some detail by H. W. Gillett in Appendix IV to this report, entitled "Discrepancies in the Load-Carrying Abilities of Carbon Steels at 850 F."

Preliminary work has been authorized with expenditures not in excess of $1500.

Project No. 19 on Procurement of Materials for Projects Nos. 10, 15, 16 and 17 (H. J. Kerr, chairman).—Coordination of many of the tests mentioned is an essential part of the present research program, requiring coordination in the preparation of materials to be tested at different laboratories. For this reason a special committee for this project was formed.

Miscellaneous Activities:

Plasticity of Metals.—The Joint Committee cooperated with the Committee on Plasticity, Division of Applied Mechanics of the American Society of Mechanical Engineers in arranging for a technical session at the December, 1936, Annual Meeting of the A.S.M.E. At that session several papers, by individual authors, and committee reports were presented, as follows:

"The Creep Curve and Stability of Steels at Constant Stress and Temperature," by S. H. Weaver,[1]
"The Interpretation of Creep Tests for Machine Design," by C. R. Soderberg,[1]
"Creep Tests at 850 F., of the 0.35 Per Cent Carbon Steel, Material K20," Report of Subgroup E of Committee III, Joint Research Committee, by C. L. Clark,[1]
"Progress Report on Long-Time Creep Tests of 18 Per Cent Chromium, 8 Per Cent Nickel Steel (K19) and 0.35 Per Cent Carbon Steel (K20) to Joint Research Committee," by H. C. Cross and J. G. Lowther,[2]
"Constant Strain Rate Tests on 0.35 Per Cent Carbon Steel (K20) at 850 F.," by A. Nadai and E. A. Davis,[2]
"A Relaxation Test on K20 Carbon Steel," by E. L. Robinson,[2] and
"A Note on Relaxation Tests at 850 F., on 0.35 Per Cent Carbon Steel, Material K20," by N. L. Mochel.[2]

Future Work:

The plan of research outlined can be expected to require the expenditure of most of the funds now available to the committee by the end of the calendar year 1938, but the plan was laid down with the view to flexibility so as to permit changes, extensions or curtailments as progress is made and a clearer view of requirements may be uncovered, or as new and timely problems are presented.

Respectfully submitted on behalf of the Joint Committee,

H. J. FRENCH,
Chairman.
N. L. MOCHEL,
Vice-Chairman.

J. W. BOLTON,
Secretary.

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[1] *Transactions*, Am. Soc. Mechanical Engrs., November, 1936.
[2] *Transactions*, Am. Soc. Mechanical Engrs., July, 1937.

APPENDIX I

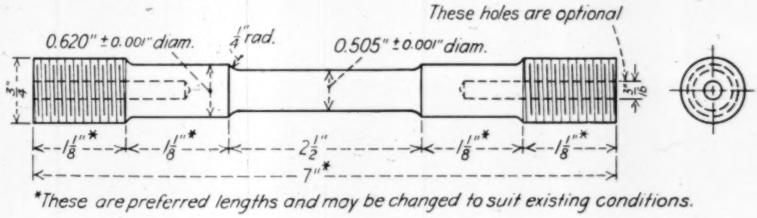
PROPOSED REVISION OF TENTATIVE METHOD OF TEST FOR SHORT-TIME HIGH-TEMPERATURE TENSION TESTS OF METALLIC MATERIALS (E 21 - 34 T)[1]

Section 3.—Change the first sentence in this section from its present form: namely,

> The tension test specimen shall be of the usual type for 2-in. gage length, see Fig. 1, as described in Section 15 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials.

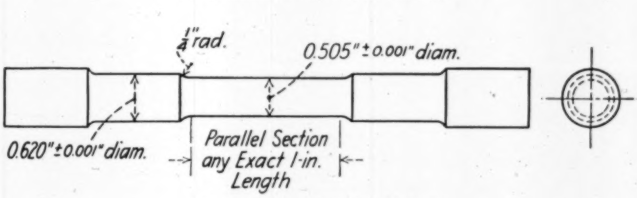
to read as follows:

> The tension test specimen shall be either of the two types shown in Fig. 1 (*a*) and (*b*). When the type shown in Fig. 1 (*b*) is used, the specimen shanks must extend beyond the furnace before gripping.


These holes are optional
0.620" ± 0.001" diam.
1/4" rad.
0.505" ± 0.001" diam.
3/4"
3/16"
1 1/8"*
1 1/8"*
2 1/2"
1 1/8"*
1 1/8"*
7"*

*These are preferred lengths and may be changed to suit existing conditions.

(*a*) Specimen with Threaded Ends.


1/4" rad.
0.505" ± 0.001" diam.
0.620" ± 0.001" diam.
Parallel Section any Exact 1-in. Length

(*b*) Specimen with Blank Ends.

FIG. 1.—Test Specimen for High-Temperature Tension Test.

Figure 1.—Delete the present Fig. 1, showing the test specimen for high-temperature tension tests, and replace with the accompanying Fig. 1 (*a*) and (*b*).

Figure 2 and Form I.—Delete Fig. 2, showing the test specimen, and Form I, giving the form for reporting temperature survey of furnace, and replace with the accompanying Fig. 2 and Form I.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 1214 (1934); also 1936 Book of A.S.T.M. Tentative Standards, p. 1307.

Section 5.—Change Paragraph (*b*) of this section to read as follows by the addition of the italicized words and symbols and the omission of the words in brackets:

(*b*) The design of the furnace shall be such that the maximum [variation of temperature over the gage length of the calibration bar] *temperature difference between any two of the points* T_i *or* T_o, T_f, C_o, B_f, *and* B_i *or* B_o, shown in Fig. 2 (see Form I), will not exceed 10 F. for temperatures up to and including 1600 F. and will not exceed 20 F. for higher temperatures.

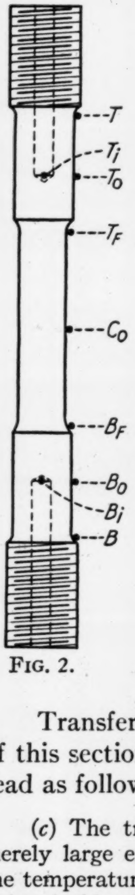


FIG. 2.

Location of Thermocouples

T At end of thread
T_i Inside of hole ½" beyond fillet
T_O Outside ½" beyond fillet
T_F At fillet
C_O Outside center of gage length
B_F At fillet
B_O Outside ½" beyond fillet
B_i Inside of hole ½" beyond fillet
B At end of thread

	Nominal Test Temperature, deg. Fahr.									
T										
T_i										
T_O										
T_F										
C_O										
B_F										
B_O										
B_i										
B										

FORM I.—Form for Reporting Temperature Survey.

Transfer the first three sentences of the Note following Paragraph (*b*) of this section to appear as a new Paragraph (*c*) of Section 5 and revise to read as follows:

(*c*) The transverse dimensions of the furnace should be as small as possible, merely large enough to allow for the shackles and extensometer. Equalization of the temperature over the length of the test specimen shall be obtained by a metallic lining inside the furnace tube, or by the use of multiple heating coils, or external shunts or other adequate means of compensation.

The remaining four sentences of the present Note are to be retained as a Note at the end of Section 5.

Section 6.—Omit Paragraph (*b*) of this section which reads as follows, designating the present Paragraph (*a*) as Section 6:

(*b*) The hot junction of the thermocouple shall be located at any one of locations T, T_0, C_0, B_0, B shown in Fig. 2, and the location of the junction shall be reported with the results of tests. Where external contact is made between thermocouple bead and the specimen, the thermocouple shall be shielded in like manner in the tension tests and in the calibration.

Section 7.—Change Paragraphs (*a*) and (*b*) of this section to read as follows by the addition of the italicized words and the omission of the words and figures in brackets:

7. (*a*) A survey of the temperature distribution throughout the test specimens shall be made before making a series of tests of specimens of a given metal, on each furnace and test shackle arrangement used. This survey shall be made using [a special calibration bar, shown in Fig. 2 (Form I), and for each series of tests the calibration bar used shall be of the same dimensions and shall be of the same type chemical composition as the test specimens to be used] *the coupons that are to be tested.* A survey of temperature distribution shall be made for each temperature at which specimens shall be tested, and detailed results of these surveys are to be reported with the results of specimen tests. These surveys furnish data for the [comparison of specimen temperature at the point where the thermocouple is applied in routine tests of specimens with the temperature of the test specimen at the middle of the reduced section of the test specimen on the axis of the specimen (C, Fig. 2)] *study of specimen and furnace temperatures.* The temperature [of the calibration bar] *during the survey* shall be also determined at [the other] *all* points indicated in Fig. 2.

(*b*) During a survey of temperature distribution [on a calibration], all temperature readings shall be taken as nearly simultaneously as possible.

Section 8.—Add a new section as Section 8 to read as follows, renumbering the remaining sections accordingly:

8. *Temperature Control During Tension Test:* (*a*) The temperature of the specimen during the actual tension test shall be measured and recorded. In all cases, a thermocouple shall be located at point C_0, and it is preferred that thermocouples be also located at point T_0 or T_1 and at point B_0 or B_1; these locations are as shown in Fig. 2. Where external contact is made between thermocouple bead and the specimen, the thermocouple shall be shielded in like manner in the tension tests and in the calibration.

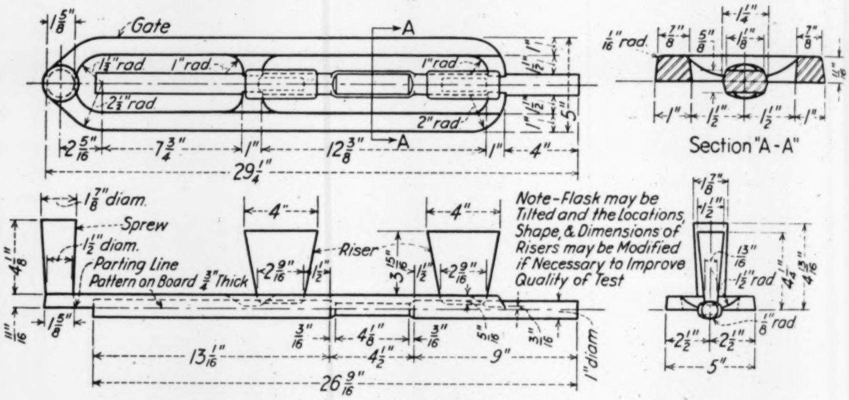
(*b*) The change in temperature over the gage length throughout the tension test should be limited to ±10 F. from the nominal test temperature.

APPENDIX II

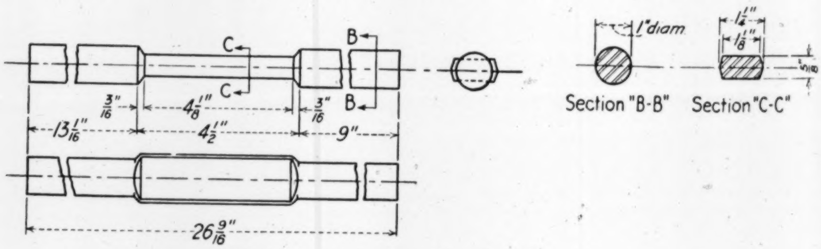
PROPOSED REVISION OF TENTATIVE METHOD OF TEST FOR SHORT-TIME HIGH-TEMPERATURE TENSION TESTS OF METALLIC MATERIALS (E 21 - 34 T)[1]

The following sections are proposed for inclusion as an Appendix to the Tentative Methods E 21 - 34 T.

A1. *Scope.*—This appendix covers a method of preparation of cast tension specimens of heat-resistant metals (chromium-nickel, chromium-nickel-iron, and chromium irons) intended for short-time tension tests up to 2200 F.


Gate
A
A
Section "A - A"
rad.
Sprew
Parting Line
Pattern on Board $\frac{3}{4}$" Thick
Riser
Note-Flask may be Tilted and the Locations, Shape, & Dimensions of Risers may be Modified if Necessary to Improve Quality of Test
$1\frac{7}{8}$" diam.
$1\frac{1}{2}$" diam.
1" diam.
$29\frac{1}{4}$"
$26\frac{9}{16}$"
$13\frac{1}{16}$"
$4\frac{1}{2}$"
9"
4"

(*a*) Details of Pattern Arrangement for Casting Tension Specimen.


C
C
B
B
1" diam.
Section "B-B"
Section "C-C"
$\frac{3}{16}$"
$4\frac{1}{8}$"
$13\frac{1}{16}$"
$4\frac{1}{2}$"
9"
$26\frac{9}{16}$"

(*b*) Cast Tension Test Specimen.

FIG. A1.—Details of Casting Tension Test Specimen for Heat-Resistant Metals.

A2. *Casting Pattern.*—Structural details of the tension specimen and pattern are given in Fig. A1. The total length of the specimen, Fig. A1 (*a*), is $26\frac{9}{16}$ in. and it is 1-in. round. The center $4\frac{1}{2}$-in. section is rectangular in shape, being $1\frac{1}{4}$ in. in width and $\frac{5}{8}$ in. in thickness.

The gates and risers are so placed with respect to this section as to insure a high degree of soundness. The particular design shown under Fig. A1 (*b*) has proven satisfactory for 35–15 nickel-chromium-iron alloys.[1] Changes in the gates and risers may be advisable for other types of alloys. The soundness of the resulting castings shall be verified by radiographic examination.

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[1] For an explanation of this proposed revision, see p. 168.

A3. *Casting Specimen.*—The gage section of the test specimen shall be machined from the central rectangular portion of the casting. Its dimensions shall conform to those given in Fig. 1 of this method. The shanks of the cast specimens are purposely made of sufficient length to remove all threaded or other connections from the heated zones of the test furnace. This procedure is necessary since specimens of this type will in general be tested at temperatures up to 2200 F. The ends of the shanks may be threaded to fit the adapters of the testing machine. The specimen shall be machined true and axial.

A4. *Testing Apparatus and Procedure.*—All details as to the tension machine and test procedure shall be in accordance with those specified in this method.

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[1] Tests by three foundries have shown that sound castings were produced consistently through the use of this pattern. Tension tests conducted by three laboratories at 1600 F. on these castings showed that the physical properties were in satisfactory agreement.

APPENDIX III

REPORT ON LONG-TIME CREEP TESTS OF 18 PER CENT CHROMIUM, 8 PER CENT NICKEL STEEL AND 0.35 PER CENT CARBON STEEL

REPORT TO THE JOINT COMMITTEE COVERING A SPONSORED RESEARCH PROJECT CARRIED OUT AT BATTELLE MEMORIAL INSTITUTE

This report is a continuing progress report on the same long-time creep tests previously reported to the sponsor societies.[1,2,3]

This program was undertaken to procure data for comparisons of creep rates over short and long periods of time. These data will permit comparisons of values obtained by extrapolations and computations from creep tests of short periods of time with the actual test data obtained over longer test periods.

Material:

The 18 per cent chromium, 8 per cent nickel steel was water quenched from 2000 F. prior to test while the 0.35 per cent carbon steel was supplied full annealed.

Details of the preparation, heat treatment, and properties of the 18 per cent chromium, 8 per cent nickel steel and the 0.35 per cent carbon steel are given in previous reports to the A.S.M.E.–A.S.T.M. Joint Research Committee on Effect of Temperature on the Properties of Metals.[4,5]

Creep Tests on 18 per cent Chromium, 8 per cent Nickel Steel (K19):

Table I gives a summary of the creep test data. The complete time-deformation curves for specimen No. B12–4 were shown in a previous progress report.[3] The time-deformation curves for specimen No. B12–1 are shown in Fig. 1. The rate of deformation is shown to have slowly but steadily increased since the last report was submitted at a test time of 15,185 hr.

At 16,447 hr., the load on specimen No. B12–1 was removed but the temperature was maintained at 1200 F. The rate of deformation at this time was about 0.00039 per cent per hr. and the total deformation was about 4.382 per cent.

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[1] H. C. Cross and F. B. Dahle, "Long-Time Creep Tests of 18 Cr 8 Ni Steel and 0.35 per cent Carbon Steel," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 58, No. 2, p. 91 (RP-58-2) (1936).

[2] Report on Long-Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel and 0.35 per cent Carbon Steel, *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 143 (1936).

[3] H. C. Cross and J. G. Lowther, *Transactions*, Am. Soc. Mechanical Engrs., Vol. 59, No. 5, p. 441, RP-59-5, July (1937).

[4] H. C. Cross, "High Temperature Tensile, Creep and Fatigue of Cast and Wrought High- and Low-Carbon 18 Cr 8 Ni Steel from Split Heats," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 56, p. 533 (RP-56-6) (1934).

[5] Short-Time Tensile Tests at 850 F. of the 0.35 per cent Carbon Steel Material K20," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 58, No. 2, p. 97 (RP-58-4) (1936).

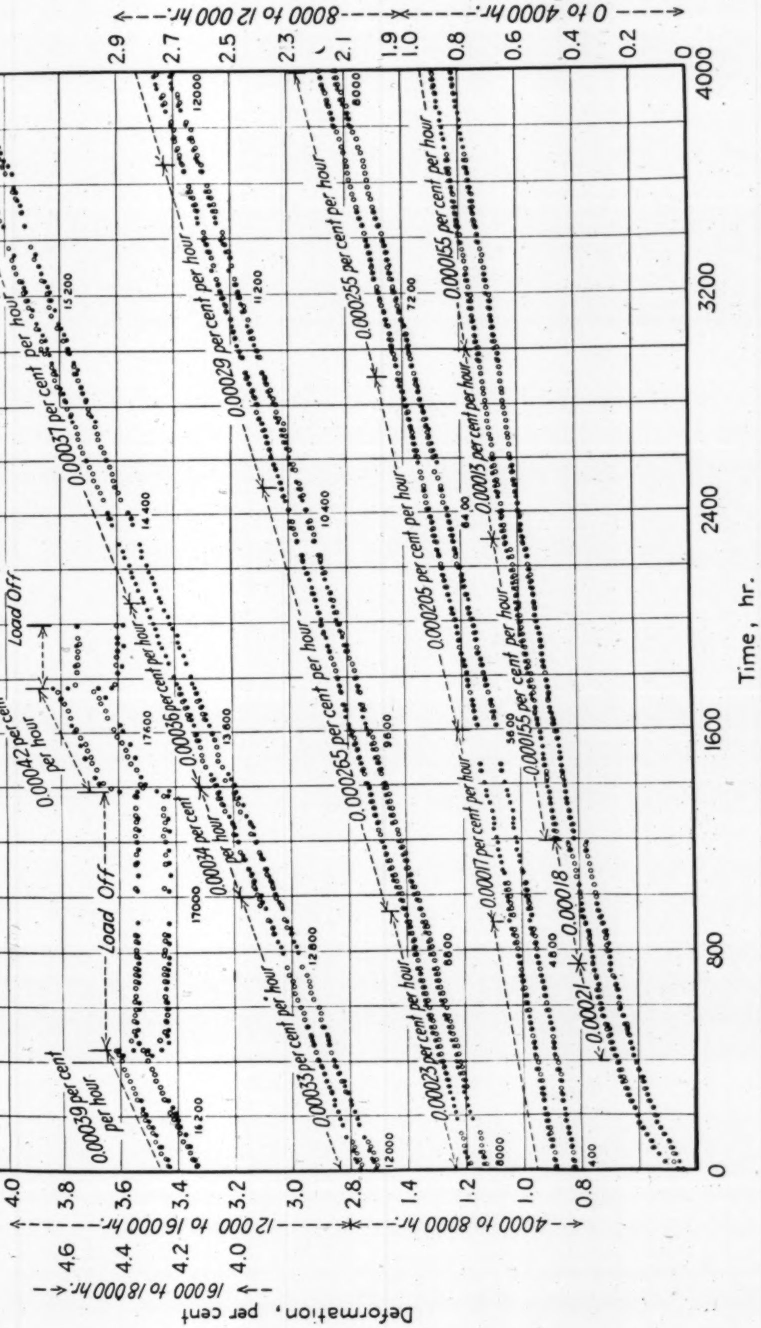

FIG. 1.—Time-Deformation Curves at 1200 F. for 18 per cent Chromium, 8 per cent Nickel Steel (K19), Specimen No. B12-1.

At the start of the test on specimen No. B12–1, the deformation resulting from application of the load was 0.06 per cent. The immediate contraction upon release of the load was 0.05 per cent and during the 936 hr. at temperature under zero load, further contraction to a total of 0.098 per cent resulted. At 17,383 hr. the load of 8345 lb. per sq. in. was reapplied and the resultant deformation was 0.053 per cent. After maintaining the load for 611 hr. to a total time of 17,994 hr., during which time the rate of deformation was about 0.00042 per cent per hr., the load was removed, resulting in a contraction of 0.052 per cent and the test was then discontinued.

TABLE I.—CREEP TEST DATA FOR 18 PER CENT CHROMIUM, 8 PER CENT NICKEL STEEL (K19).

Test Temperature 1200 F.; Load 8345 lb. per sq. in.

	Specimen No. *B*12–4		Specimen No. *B*12–1	
Initial Deformation, per cent....	0.06		0.06	
Duration of Test, hr............	10,127		16,447	
	Rate of Deformation, per cent per hr.	Total Deformation, per cent	Rate of Deformation, per cent per hr.	Total Deformation, per cent
500 hr....................	0.00043	0.41	0.00021	0.27
1000 hr....................	0.00023	0.54	0.00018	0.36
1600 hr....................	0.00021	0.67	0.000155	0.50
2000 hr....................	0.00021	0.75	0.000155	0.57
3000 hr....................	0.00021	0.96	0.00013	0.70
4000 hr....................	0.00021	1.16	0.000155	0.86
5000 hr....................	0.000255	1.45	0.00017	1.01
6000 hr....................	0.000255	1.63	0.000205	1.19
7000 hr....................	0.000285	1.92	0.00021	1.40
8000 hr....................	0.00028	2.19	0.000255	1.66
9000 hr....................	0.000365	2.49	0.000265	1.89
10000 hr....................	0.000475	2.899	0.00026	2.11
11000 hr....................	....	See Note 1	0.00029	2.44
12000 hr....................	....	.....	0.000325	2.74
13000 hr....................	....	.....	0.000335	3.081
14000 hr....................	....	.....	0.000355	3.442
15000 hr....................	....	.....	0.00036	3.807
16000 hr....................	....	.....	0.00039	4.178 See Note 2

NOTE 1.—At 10,127 hr. and 2.962 per cent deformation:
Immediate contraction upon release of load, 0.048 per cent.
Greatest contraction during 645 hr. at temperature under zero load, 0.071 per cent.
Immediate deformation upon reapplication of load at 10,772 hr., 0.045 per cent.
Load on for 695 hr., load off at 11,467 hr., immediate contraction upon release of load, 0.047 per cent.
Test discontinued.

NOTE 2.—At 16,447 hr. and 4.382 per cent deformation:
Immediate contraction upon release of load, 0.05 per cent.
Greatest contraction during 936 hr. at temperature under zero load, 0.098 per cent.
Immediate deformation upon reapplication of load at 17,383 hr., 0.053 per cent.
Load on for 611 hr., rate 0.00042 per cent per hr.
Load off at 17,994 hr.
Immediate contraction upon release of load, 0.052 per cent.
Test discontinued.

It may be seen that the deformations and contractions resulting from application of the load at the start of the test and release and reapplication of the load after considerable deformation took place are about the same magnitude. It may also be seen that only about 0.048 per cent additional contraction to the 0.05 per cent measured upon release of the load took place during the 936 hr. at temperature under no load. This value of

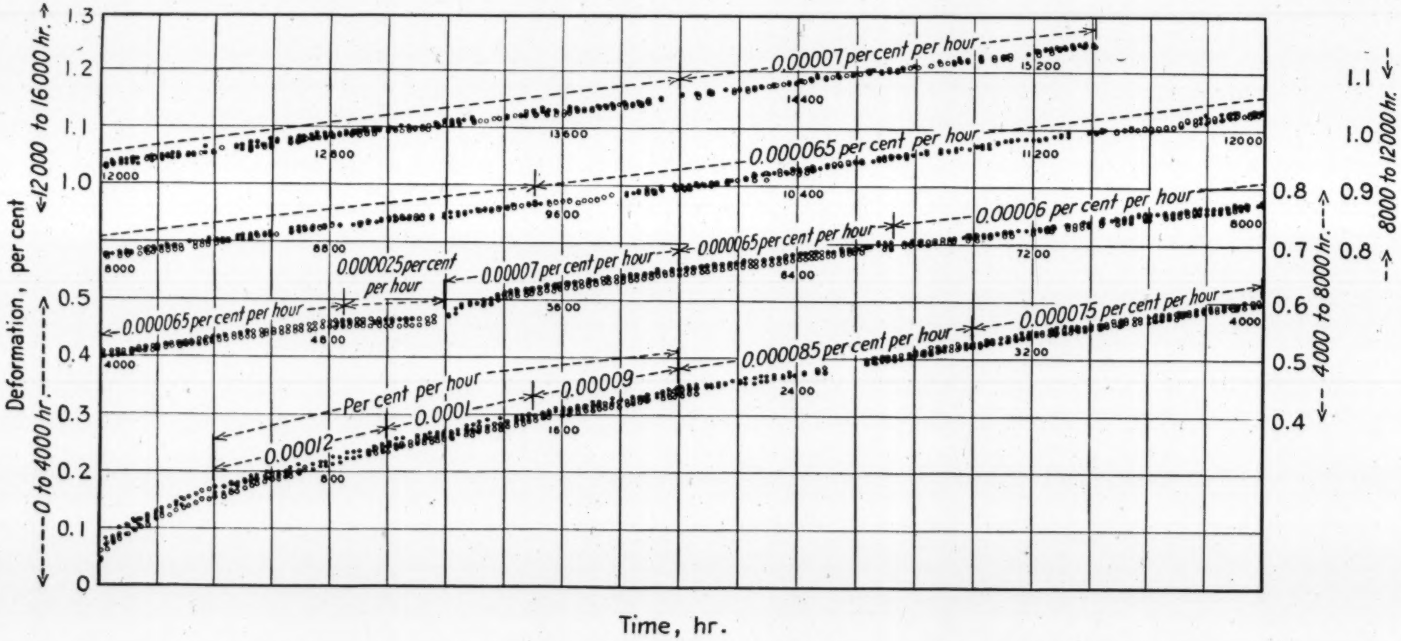

FIG. 2.—Time-Deformation Curves at 850 F. for 0.35 per cent Carbon Steel (K20), Specimen No. 25B-5.

0.048 per cent is very small when compared with the total deformation of 4.382 per cent measured during the 16,447 hr. under load.

The test data on loading and unloading of both specimens Nos. B12-4 and B12-1 indicate that practically all of the deformation subsequent to that measured upon application of the load is permanent and not recoverable for an 18 per cent chromium, 8 per cent nickel steel tested at 1200 F. and 8345 lb. per sq. in.

TABLE II.—CREEP TEST DATA FOR 0.35 PER CENT CARBON STEEL (K20).

Test Temperature 850 F.

	Specimen No. 10A-4		Specimen No. 25B-5	
Load, lb. per sq. in.	8 000		7 500	
Initial Deformation, per cent	0.04		0.05	
Duration of Test, hr	9 617		15 400	
	Rate of Deformation, per cent per hr.	Total Deformation, per cent	Rate of Deformation, per cent per hr.	Total Deformation, per cent
500 hr	0.00017	0.21	0.00012	0.175
1000 hr	0.00015	0.28	0.00012	0.24
1400 hr	0.00013	0.34	0.00010	0.28
2000 hr	0.00011	0.40	0.00009	0.34
3000 hr	0.00010	0.51	0.00009	0.425
4000 hr	0.00009	0.605	0.000075	0.50
5000 hr	0.000075	0.688	0.000025	0.56
6000 hr	0.000075	0.763	0.00007	0.60
7000 hr	0.000075	0.844	0.00006	0.712
8000 hr	0.000085	0.919	0.00006	0.773
9000 hr	0.000095	1.015	0.00006	0.839
10000 hr	....	See Note 1	0.000065	0.899
11000 hr	....	.....	0.000065	0.969
12000 hr	....	.....	0.000065	1.037
13000 hr	....	.....	0.000065	1.095
14000 hr	....	.....	0.000065	1.155
15000 hr	....	.....	0.00007	1.225
16000 hr	....	.....	....	See Note 2

NOTE 1.—At 9617 hr. and 1.173 per cent total deformation:
Immediate contraction upon release of load, 0.039 per cent.
Greatest contraction during 643 hr. at temperature under zero load, 0.064 per cent.
Immediate deformation upon reapplication of load at 10,260 hr., 0.045 per cent.
Load on for 695 hr., load off at 10,955 hr., immediate contraction upon release of load, 0.043 per cent.
Test discontinued.

NOTE 2.—Test continuing, rate 0.00007 per cent per hr. and deformation:
1.25 per cent at 15,400 hr.

Creep Tests on 0.35 per cent Carbon Steel (K20):

Table II gives a summary of the creep test data. The complete time-deformation curves for specimen No. 25B-5 are shown in Fig. 2. The complete time-deformation curves for specimen No. 10A-4 were shown in a previous progress report.[1]

The test on specimen No. 25B-5 is still in progress at 15,400 hr. with a rate of deformation of about 0.00007 per cent per hr. and a total deformation of about 1.25 per cent. There has been only a slight increase in rate since the last report at 12,214 hr. from 0.000065 to 0.00007 per cent per hr.

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[1] H. C. Cross and J. G. Lowther, *Transactions*, Am. Soc. Mechanical Engrs., Vol. 59, No. 5, p. 441, RP-59-5, July (1937).

Discussion of the Results of the Creep Tests:

For greater ease of comparison the time-deformation curves for the four specimens tested are shown as single curves in Fig. 3.

Since the rate of deformation for specimen No. B12–1 (18 per cent chromium, 8 per cent nickel steel) was still increasing at the termination of the test, the difference was increasing steadily between the actual deformation measured and the value that would be obtained by extrapolation from shorter and more usual test periods such as 2000 hr.

The relatively small effect of unloading and reloading of specimens Nos. B12–4 and B12–1 should be noted.

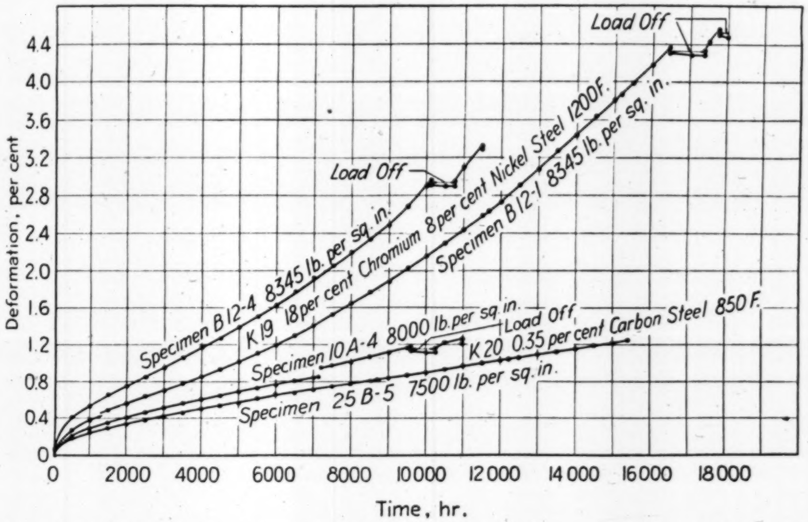

FIG. 3.—Time-Deformation Curves for 18 per cent Chromium, 8 per cent Nickel Steel (K19) at 1200 F. and 0.35 per cent Carbon Steel (K20) at 850 F.

Although the rate of deformation for specimen No. 25B–5 (0.35 per cent carbon steel) has changed very little over the last 8000 hr., since its rate subsequent to the rate at 2000 hr. has been lower, extrapolation from 2000 hr. leads to differences less than the actual measured deformation and therefore to differences on the conservative side. Only continuation of the test period for this carbon-steel specimen will indicate the extent of time over which an approximately constant rate will be obtained or whether the rate will further decrease or increase.

Apparently, the load of 7500 lb. per sq. in. at 850 F. for this 0.35 per cent carbon steel (K20) is just on the border line. Lower stresses would likely produce more pronounced strain-hardening with consequent reduction of the creep rate while higher stresses of 8000 lb. per sq. in. (see Fig. 3 for specimen No. 10A–4) or more, would considerably shorten the period over which the rate is constant or changing slowly and increasing rates would be more quickly shown.

These data further confirm the conclusion that for dependable extrapolations, knowledge of the strain-hardening characteristics of the materials is absolutely necessary.

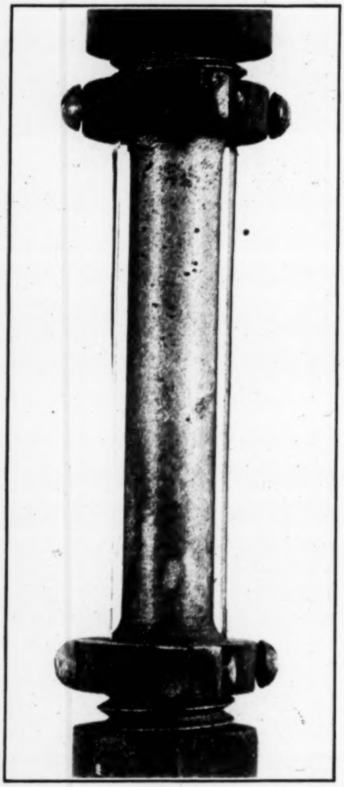

FIG. 4.—External Appearance of Specimen No. B12–1 (18 per cent Chromium, 8 per cent Nickel Steel, K19) after removal from Creep Test of 17,994 hr. at 1200 F.

Examination of Specimens After Creep Test:

Surface.—Figure 4 shows the external appearance of specimen No. B12–1 (18 per cent chromium, 8 per cent nickel steel) as removed from the creep test unit with the platinum measuring strips still in place after 17,994 hr. at 1200 F. at a load of 8345 lb. per sq. in. The specimen shows practically no scale and was quite evenly discolored, checking the appearance of specimen No. B12–4 removed after 11,467 hr. at 1200 F. but differing from a previous specimen which was removed due to excessive rate of deformation and which showed a spotty appearance.

Structure.—Figure 5 shows the structure of a longitudinal section taken from the center of the creep test specimen No. B12–1 (18 per cent chromium, 8 per cent nickel steel) after test of 17,994 hr. at 1200 F. Carbide precipitation has taken place in the grain boundaries, along twinning planes, and also within the grains themselves. Considerable agglomeration of the carbide particles has apparently taken place. No difference in structure between the center and surface was noted, nor was any intergranular parting or oxidation observed at the surface.

The structure for this specimen No. B12–1 was very similar to that observed for specimen No. B12–4 and shown in a previous report.[1]

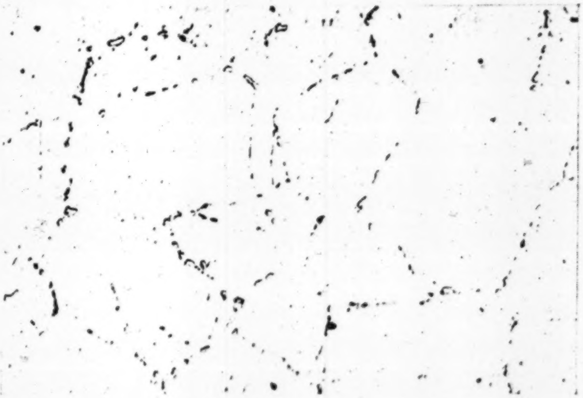

FIG. 5.—Structure of Longitudinal Section of Specimen No. B12–1 (18 per cent Chromium, 8 per cent Nickel Steel, K19) after 17,994 hr. at 1200 F. (× 500.)

Etchant, aqua regia in glycerine

Impact Resistance.—Two round Izod impact test specimens (V-notch type) were machined from the reduced section of creep test specimen No. B12–1. The results obtained from the impact test were as follows:

	IZOD IMPACT RESISTANCE, FT-LB. (ROUND 0.45 IN. DIAM., "V"-NOTCH SPECIMENS)
18 per cent Chromium, 8 per cent Nickel Steel:	
As heat treated	101 to 107
Specimen No. B12–4 (11,467 hr. at 1200 F.)	50 to 58
Specimen No. B12–1 (17,994 hr. at 1200 F.)	32 to 35

The longer exposure at 1200 F. for specimen No. B12–1 (17,994 hr.) as compared with specimen No. B12–4 (11,467 hr.) has resulted in a further reduction in impact resistance to 32 to 35 ft-lb. While a value of 32 to 35 ft-lb. shows a considerable drop from the initial value as heat treated, the steel still shows considerable, and for most purposes ample, toughness.

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[1] H. C. Cross and J. G. Lowther, *Transactions*, Am. Soc. Mechanical Engrs., Vol. 59, No. 5, p. 441, RP-59-5, July (1937).

If this reduction in impact resistance continued to progress at this rate, it would be practically lost at 30,000 hr.

Comparison of the structure of specimens Nos. B12–4 and B12–1 give no hint of the further reduction of the impact resistance of specimen No. B12–1. In fact, agglomeration of the carbides in specimen No. B12–1 suggested before the tests that a similar or even higher impact resistance might be expected.

Conclusions:

The long-time creep tests on the 18 per cent chromium, 8 per cent nickel steel at 8345 lb. per sq. in. at 1200 F. showed increasing rates of creep, thereby increasing the difference between the actual deformations measured and the values obtained by extrapolation from the 2000-hr. test period.

The long-time creep tests on the 0.35 per cent carbon steel at 8000 and 7500 lb. per sq. in. at 850 F. continue to indicate extrapolation from the 2000-hr. period as conservative as compared with the actual deformation measured.

The data further confirm the conclusion that for dependable extrapolations, knowledge of the strain-hardening characteristics of the materials is essential.

Experiments on both the 18 per cent chromium, 8 per cent nickel steel and the 0.35 per cent carbon steel on unloading, holding at temperature with zero load, reloading, and again unloading indicate that practically all of the deformation subsequent to that measured upon application of the load at the start of the test is permanent and not recoverable.

Respectfully submitted on behalf of the committee,

HOWARD C. CROSS,
J. G. LOWTHER.

APPENDIX IV

DISCREPANCIES IN THE LOAD-CARRYING ABILITIES OF CARBON STEELS AT 850 F.

By H. W. Gillett[1]

Notwithstanding the urge for the use of higher temperatures and the development of alloy steels to serve at higher temperatures, plain carbon steel, for the sake of economy, is used in steam plants, turbines, oil stills and in similar applications for high-temperature service within its capabilities. Since 850 F. seems, in many types of service, to be within the proper temperature range for use of carbon steel, that temperature is of particular importance, hence most studies of carbon steel for high-temperature service either include this temperature or bracket it.

Designers have set up figures for acceptable working stresses at such temperatures. Tapsell[2] cites the proposals of Jacobus, Jasper, Hatfield, and himself for medium carbon steel at 450 C. (842 F.) as ranging from 7000 to 9000 lb. per sq. in. Dixon[3] cites an oil refiner whose design allows 7000 lb. per sq. in at 850 F. for 0.20 to 0.30 per cent carbon steel.

Such working stresses are based on the creep characteristics, after allowing a suitable margin of safety. A turbine builder will adopt a lower working stress than an oil refiner. Allen[4] places 6000 lb. per sq. in. at 800 F. as the limit for turbine cylinders of 0.28 to 0.37 per cent carbon-steel castings.

There are carbon steels whose creep properties obviously allow an ample factor of safety under such design stresses. There are others which do not. To simplify consideration, the present discussion will be confined to the behavior of normalized and annealed steels at 850 F. Rimming or semikilled steel is recognized as having much poorer creep properties than killed steel, as Kinzel[5] stated, and as White and Clark[6] showed.

White and Clark cited a steel of 0.18 per cent carbon, 0.49 per cent manganese, and 0.01 per cent silicon, which showed a load of 10,000 lb. per sq. in. for 1.0 per cent final creep rate in 1000 hr. at 850 F., while an 0.41 per cent carbon, 0.92 per cent manganese, and 0.21 per cent silicon killed steel gave 16,750 lb. per sq. in.

Jenkins, Tapsell, Mellor and Johnson[7] cite a normalized rimmed steel of 0.16 per cent carbon, 0.46 per cent manganese, 0.006 per cent silicon, made in a basic open-hearth furnace, which, loaded at 17,900 lb. per sq. in. (8 tons per sq. in.) stretched

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[1] Metallurgist, Battelle Memorial Inst., Columbus, Ohio.

[2] H. J. Tapsell, "Creep of Metals," Oxford University Press, London (1931).

[3] E. S. Dixon, "Needs of the Oil Industry for Metals at High Temperatures," Symposium on Effect of Temperature on the Properties of Metals, published by the American Society for Testing Materials and The American Society of Mechanical Engineers, p. 66 (1931). Symposium available as separate publication.

[4] R. C. Allen, "Steam Turbine Materials for High Temperatures," Symposium on Effect of Temperature on the Properties of Metals, published by the American Society for Testing Materials and The American Society of Mechanical Engineers, p. 30 (1931). Symposium available as separate publication.

[5] Discussion by A. B. Kinzel of paper by A. P. Spooner and F. B. Foley, "High-Temperature Creep Properties of Wrought Carbon and Low-Alloy Steels," Symposium on Effect of Temperature on the Properties of Metals, p. 389, published by the American Society for Testing Materials and The American Society of Mechanical Engineers (1931). Symposium available as separate publication.

[6] Discussion by A. E. White and C. L. Clark, of paper by A. P. Spooner and F. B. Foley, "High-Temperature Creep Properties of Wrought Carbon and Low-Alloy Steels," Symposium on Effect of Temperature on the Properties of Metals, p. 386, published by the American Society for Testing Materials and The American Society of Mechanical Engineers (1931). Symposium available as separate publication.

[7] C. H. M. Jenkins, H. J. Tapsell, G. A. Mellor, and A. E. Johnson, "Some Aspects of the Behavior of Carbon and Molybdenum Steels at High Temperatures," Preprint for the Third World Power Conference held in Washington, D. C., September 7 to 12, 1936.

7 per cent in 480 hr. at 842 F., while another normalized steel of 0.15 per cent carbon, 0.76 per cent manganese, 0.28 per cent silicon made in an acid open-hearth furnace under the same conditions stretched but 0.17 per cent, that is, only $\frac{1}{400}$ as much. At the end of the 480 hr. the rimmed steel was continuing to stretch 134 times as fast.

Such data give ample reason for specifying killed steels for high-temperature service. However, killed steels, too, may vary.

Clark and White[1] give the following comparison for steels annealed at 1550 F.:

	Carbon, per cent	Manganese, per cent	Silicon, per cent	McQuaid-Ehn Grain Size	Stress for Final Creep Rate of 0.10 per cent in 1000 hr., lb. per sq. in.		
					800 F.	900 F.	1000 F.
Steel A, open hearth	0.15	0.46	0.28	5 to 6	17 200	......	3300
Steel B, electric furnace	0.15	0.50	0.23	4 to 5	26 800	16 900	5750

E. L. Robinson has studied the available data for Clark and White's steel B, for an annealed basic open-hearth steel of 0.18 per cent carbon, 0.51 per cent manganese, and 0.09 per cent silicon reported on by Babcock and Wilcox,[2] and for an annealed basic open-hearth steel (K20) cooperatively tested by the Joint Committee, which contains 0.35 per cent carbon, 0.55 per cent manganese, and 0.19 per cent silicon. Interpolating for the first two steels he concludes that for a final creep rate of 0.1 per cent in 1000 hr. at 850 F. the steels compare as follows:

	PROCESS	MCQUAID-EHN GRAIN SIZE	CREEP LOAD, LB. PER SQ. IN.
Clark and White Steel B	Electric arc	4 to 5	23 500
Babcock and Wilcox Steel	Basic open hearth	6	14 500
Steel K20	Basic open hearth	8	7 500

Cross and Dahle[3] studied two forged steels LF and F, with the following results:

	Carbon, per cent	Manganese, per cent	Silicon, per cent	Aluminum Used, lb. per ton	McQuaid-Ehn Grain Size	Total Deformation in 1300 hr. (Load 12,000 lb. per sq. in. at 850 F.), per cent	Final Rate of Creep, per cent per hr.
Steel LF	0.27	0.63	0.18	0.45	2 to 4 (coarse)	0.155	0.00002
Steel F	0.37	0.61	0.24	0.80	6 to 8 (fine)	0.50	0.00020

The room temperature and the short-time tensile and yield strengths at 850 F. of steel LF were slightly below those of steel F, yet at the end, steel LF was stretching but $\frac{1}{10}$ as much as steel F. Both steels were poured into the same size ingots and both were tested in the normalized condition.

White and Clark[1] comment that electric-arc melted steel appears more creep resistant than basic open-hearth steel in their tests, also that induction-melted steel may be more resistant than arc-melted steel, qualifying these statements by pointing out that melting practice may differ even with the use of the same melting medium. They also comment that ingot size and degree of reduction in hot working may be important variables.

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[1] C. L. Clark and A. E. White, "The Properties of Metals at Elevated Temperatures," *Engineering Research Bulletin*, No. 27, March, 1936.

[2] Digest of Properties, Carbon and Alloy Steels for Cracking Still Tubes, 1935 Revision of *Technical Bulletin No. 6a*, Babcock and Wilcox Tube Co.; see also *Metals and Alloys*, Vol. 7, No. 3, March, 1936, p. 73.

[3] H. C. Cross and F. B. Dahle, "High-Temperature Properties of Cast and Wrought Carbon Steels from Large Valves for High Temperature Service," *Transactions*, Am. Soc. of Mechanical Engrs., February, 1936, p. 103.

Jenkins, Tapsell, Mellor and Johnson[1] studied two series of steels, a low-carbon series normalized from 1740 F., a medium-carbon series from 1650 F., with the following results. All were loaded at 17,900 lb. per sq. in. at 842 F. for 480 hr.

Steel	Carbon, per cent	Manganese, per cent	Silicon, per cent	Process	Deoxidation with	Total Creep, per cent on gage length	Final Creep Rate, in. per in. per hr. × 10^{-6}	
A	0.13	0.53	0.16	Basic electric furnace	0.14 lb. Al per ton	0.40	1.3	Average 0.8
F	0.15	0.76	0.28	Acid open hearth	SiMn, FeMn, 0.42 lb. Al per ton	0.17	0.8	
G[a]	0.16	0.66	0.14	Acid open hearth	FeSi, SiMn, FeMn	0.18	0.8	
H	0.20	0.56	0.20	Basic electric furnace	FeMn, 0.44 lb. Al per ton	0.19	0.3	
E	0.19	0.40	0.08	Acid open hearth	FeSi, FeMn	1.10	10.8	
D	0.37	0.73	0.31	Not known	Not known	0.14	0.4	Average 1.4
J	0.40	0.58	0.21	Acid open hearth	FeSi, FeMn	0.23	1.7	
K	0.42	0.50	0.31	Acid open hearth	FeSi, FeMn, FeTi	0.42	2.1	
L	0.40	0.53	0.16	Acid open hearth	FeMn	0.27	1.5	
M	0.44	0.55	0.14	Basic electric furnace	FeTi	0.34	1.5	
N	0.40	0.48	0.10	Acid open hearth	FeSi, FeMn	0.44	2.1	
B	0.40	0.59	0.21	Basic electric furnace	0.14 lb. Al per ton	1.28	18.5	

[a] Forged steel.

The McQuaid-Ehn carburizing test was made, without showing abnormality in any of the steels. McQuaid-Ehn grain size is not specifically indicated, but it is stated that no differences in grain size were noted in the normalized steels. Nor did oxygen content give any clue.

Unless the low silicon content gives an indication as to steel E, there is nothing in the record to explain its poor creep resistance, and in the case of steel B nothing whatever is at hand to explain its behavior.

Steel B does show that the qualifying comment made by White and Clark[1] should be applied to their observation on electric *versus* open-hearth steels, since steel B, electric melted, was the worst of the lot except the open-hearth steel previously referred to and not included in the foregoing table.

The most interesting fact brought out by Jenkins and co-workers is that when tested in the cast condition, before rolling, and annealed at 1560 F. instead of normalized, steels A and B maintained their relative position, as shown in the following table:

	CARBON, PER CENT	CONDITION	HEAT TREATMENT	FINAL RATE OF CREEP AT 480 HR. (Load of 17,900 lb. per sq. in. at 842 F.), in. per in. per hr. × 10^{-6}
Steel A	0.13	Cast	Annealed	2.7
Steel A	0.13	Rolled	Normalized	1.3
Steel B	0.40	Cast	Annealed	30.4
Steel B	0.40	Rolled	Normalized	18.5

In either state, steel B shows 11 to 14 times the rate of creep of steel A.

It has been suggested by P. E. McKinney that by a suitable grain-coarsening treatment the K20 steel of No. 8 grain size, mentioned above, might be put in a better condition to resist creep. This K20 steel was made and treated with the sole idea of obtaining uniformity, rather than creep resistance.

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[1] *Loc. cit.*

Jenkins and co-workers coarsened several normalized steels by annealing 6 hr. at 2010 F. and furnace cooling. The total strain in inches per inch after 480 hr. at their usual test conditions of 17,900 lb. per sq. in. and 842 F. was as follows:

	TOTAL STRAIN AFTER 480 HR. (Load of 17,900 lb. per sq. in. at 842 F.), in. per in.	
	NORMALIZED	COARSENED
Steel A	0.004	0.0045
Steel E	0.011	0.0125
Steel K	0.0045	0.0035
Steel L	0.003	0.004
Steel B	0.013	0.006

Steel B of low creep resistance was improved by coarsening the grain, but steel E, the other killed steel of low creep resistance, was not.

It is extremely disconcerting to the user of steel at high temperatures to know that killed steel may vary as widely from the optimum in creep resistance as do steel K20 of the Joint Committee and steels E and B of Jenkins and co-workers. He cannot find full solace in specifying electric-melted killed steel in the face of Jenkins' electric steel B. He cannot adopt some particular grain coarsening treatment with full assurance, since what helped steel B did not help steel E.

His only recourse at the moment is to subject to creep test, in the condition of heat treatment in which he expects to use it, each heat of steel and possibly each part of the heat that has been cast in different sized ingots or received different amounts of working, since the difference, as was shown by Cross and Dahle[1] is not necessarily brought out by short-time high-temperature tension tests.

One can no longer pass off these discrepancies in behavior of different lots of steel on the assumption that the steels are really alike and the differences are due to careless laboratory technique in creep testing. The cooperative work on creep testing of steel K20, reported to the committee but not yet published, is sufficient to show that any laboratory that conducts its creep tests strictly in accordance with the procedure described in the Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (A.S.T.M. Designation: E 22-35 T) of the American Society for Testing Materials[2] will differentiate sharply between the creep-resistant and the non-creep-resistant steels under discussion. There are differences in steels that are huge from the point of view of load-carrying ability at high temperatures.

The method of melting, the McQuaid-Ehn grain size, and grain-coarsening treatments all seem to bear some relation to high-temperature performance. The behavior of rimmed steel as compared with fully killed steel and the carry-over of creep resistance or its lack in both the cast and the rolled condition, also point to the grain-coarsening properties of the steel and to the melting and deoxidation practice which controls them, as worthy of attention in connection with the problem of production of creep-resistant steels.

While greater refinement in methods of creep testing and more knowledge of the effect of multidimensional stress and the like in high-temperature problems are needed, attention should not be confined to such matters to the neglect of the steelmaking variables that produce a ten-fold variation in creep behavior of killed steels and a hundred-fold variation between killed and rimmed steels.

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[1] *Loc. cit.*

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1291 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 1316.

This has long been recognized, but very mildly stated. Tapsell,[1] for example, said "A steel of a particular carbon content made by one process may be expected to differ to some extent from a steel made by another process." Now that evidence is piling up from all sides that steels may differ to a huge extent, it behooves makers and users of steel for creep-resistant service to tackle the problem so that the steel maker may know how to produce consecutive heats of steels not only of the same analysis, and having the same short-time high-temperature tension properties, but also of consistent creep properties, and so that the user may be able to count upon that consistency.

Since the statement of a problem is a necessary step toward its solution, the Joint Committee makes this presentation. It is also undertaking preliminary experimental work in the hope of opening up some possible paths toward the clarification of the problem.

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[1] *Loc. cit.*

DISCUSSION

Mr. H. J. Gough.[1]—With regard to Mr. Gillett's paper I should like to offer a few remarks on the tests made at the National Physical Laboratory quoted by him and to call attention to certain abnormalities which were encountered. In the first place, I can strongly support Mr. Gillett's opinion that these differences cannot be ascribed to changes or errors in the testing technique employed; all tests were made under identical and very carefully controlled conditions. The abnormalities encountered in certain steels and the differences in behavior between similar batches of one steel have given great concern to the members of the committee which controls the researches referred to. One case was particularly unsatisfactory. Special precautions were taken to obtain a uniform and representative batch of steel for research purposes. Its manufacture was carefully controlled and recorded through every stage. Later on, the supply ran out and it was decided to obtain a further supply of exactly the same material. Every stage of the manufacture was, therefore, repeated exactly, the fullest cooperation in our desired object being obtained from the maker. The two steels proved to be indistinguishable with regard to chemical composition, microstructure and the ordinary mechanical tests conducted at air temperature. Yet the *creep* behavior differed very widely, that of one being of a different *order* to that of the other. Every attempt has since been made to trace down the responsible factor but without success and the matter remains a puzzling and serious mystery. The unsuspected use of such an abnormal steel for certain construction, such as turbines, say, might be a serious matter. In an endeavor to guard against this possibility, attention is being given to the possibilities of the use of a relatively quick method of creep test lasting, at the most, a few days. We are quite convinced that such a test cannot predict the creep rate after a really long service history, but it might give sufficient indication of the presence of an abnormal batch of material.

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[1] Superintendent, Engineering Dept., National Physical Laboratory, Teddington, Middlesex, England.

REPORT OF COMMITTEE B-1

ON

COPPER AND COPPER ALLOY WIRES FOR ELECTRICAL CONDUCTORS

Committee B-1 on Copper and Copper Alloy Wires for Electrical Conductors held one meeting during the year, in Chicago, Ill., on March 4, 1937.

Only one change has been made in the committee personnel. Mr. J. L. Minick of the Pennsylvania Railroad, Electrical Department, has been appointed to the committee to serve with Mr. J. A. Jones as a representative of the Association of American Railroads.

The Standard Specifications for Bronze Trolley Wire (B 9 – 36) include three classes of wire of varying strengths and conductivity. It has come to the committee's attention that a large percentage of wire being purchased as Class A wire under these specifications is actually being furnished with 55 per cent conductivity instead of 40 per cent conductivity, as called for in the specifications. This 55 per cent conductivity wire meets or exceeds all other requirements of Class A wire. The committee feels that no change should be made in Standard Specifications B 9 at the present time, but that this report should record the fact that higher conductivity wire, otherwise similar, meeting the requirements of Class A wire under these specifications, is available and may be obtained from manufacturers.

There has been a request from the manufacturers of trolley wire hardware that the committee prepare standard specifications for figure 8 trolley wire. However, Committee B-1 feels that it is unable at the present time to prepare specifications for these materials due to a lack of consumer representation on the committee so far as users of figure 8 trolley wire are concerned. The committee feels, however, that it can be of service in crystallizing manufacturing practice so far as the dimensions and details of the shapes of figure 8 trolley wire sections are concerned, and recommends to the manufacturers that they consider standardizing on the shapes shown in the accompanying Fig. 1, these shapes being practically the same, with the addition of dimensional tolerances, as those given in the Standard Specifications for Hard-Drawn Copper Wire as published in 1912.[1] This is being referred to the appropriate section of the National Electrical Manufacturers Association.

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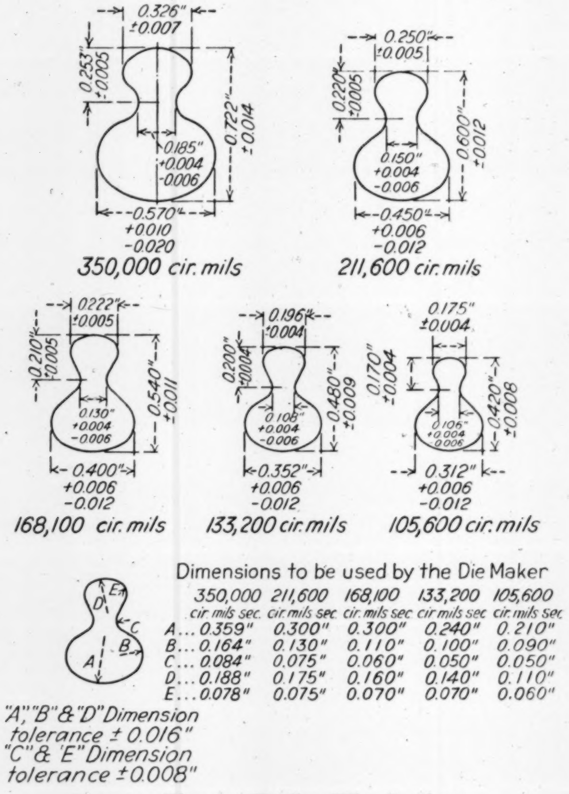
[1] *Proceedings*, Am. Soc. Testing Mats., Vol. XII, p. 271 (1912).

Both the Standard Specifications for Bronze Trolley Wire (B 9 – 36) and the Standard Specifications for Copper Trolley Wire (B 47 – 36) include grooved trolley wire sections as large as 350,000 cir. mils. Discussion with consumers' representatives brings out the fact that there will in all probability be an increasing demand for a 400,000 cir. mils grooved trolley wire to supplement the sizes called for in these specifications. The attention of the committee has also been called to the fact that there is a small demand for the so-called figure 9 deep-section grooved trolley wire which has groove and top lobe dimensions identical with the grooved sections covered by the Standard Specifications B 9 and B 47, but the bottom lobe of which is of quite different shape, being narrower and deeper in proportion. Discussion with interested groups of the National Electrical Manufacturers Association has brought out the fact that the figure 9 deep-section wire is particularly adapted for use in mines where trolley wheels are usually used, while the 400,000 cir. mils grooved trolley wire, which would supplement the sizes shown in Fig. 1 of Standard Specifications B 9 and B 47, would generally be used with sliding contacts.


0.326" ±0.007
0.253" ±0.0005
0.722" ±0.014
0.185" +0.004 -0.006
0.570" +0.010 -0.020
350,000 cir. mils
0.250" ±0.005
0.220" ±0.0005
0.600" ±0.012
0.150" +0.004 -0.006
0.450" +0.006 -0.012
211,600 cir. mils
0.222" ±0.005
0.210" ±0.005
0.540" ±0.011
0.130" +0.004 -0.006
0.400" +0.006 -0.012
168,100 cir. mils
0.196" ±0.004
0.200" ±0.004
0.480" ±0.009
0.108" +0.004 -0.006
0.352" +0.006 -0.012
133,200 cir. mils
0.175" ±0.004
0.170" ±0.004
0.420" ±0.008
0.106" +0.004 -0.006
0.312" +0.006 -0.012
105,600 cir. mils
A
B
C
D
E

Dimensions to be used by the Die Maker

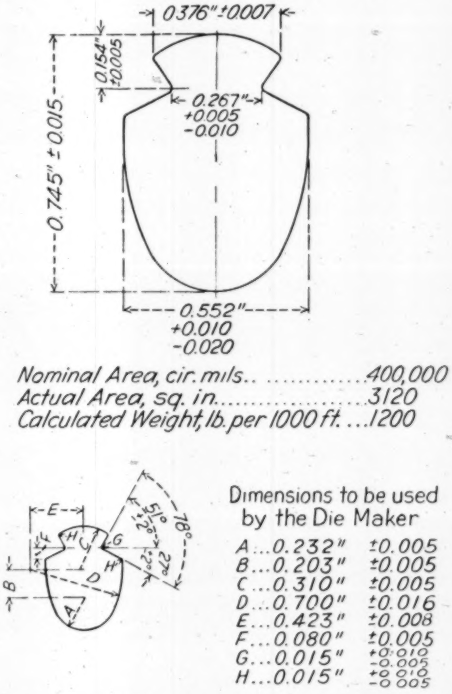
	350,000 cir. mils sec.	211,600 cir. mils sec.	168,100 cir. mils sec.	133,200 cir. mils sec.	105,600 cir. mils sec.
A...	0.359"	0.300"	0.300"	0.240"	0.210"
B...	0.164"	0.130"	0.110"	0.100"	0.090"
C...	0.084"	0.075"	0.060"	0.050"	0.050"
D...	0.188"	0.175"	0.160"	0.140"	0.110"
E...	0.078"	0.075"	0.070"	0.070"	0.060"

"A," "B" & "D" Dimension tolerance ± 0.016"
"C" & "E" Dimension tolerance ± 0.008"

FIG. 1.—Figure 8 Trolley Wire Sections.

It is the committee's recommendation that the manufacturers consider standardizing on the shape shown in the accompanying Fig. 2 for the figure 9 deep-section grooved trolley wire of 400,000 cir. mils.

Also, it is recommended that the manufacturers consider standardizing on a 400,000 cir. mils section applying to Standard Specifications B 9 and B 47, if and when the demand therefor appears, in which the upper lobe, the groove, and the lower lobe width shall have dimensions identical with the 350,000 cir. mils sections of Fig. 1 of these specifications,[1] increasing the depth of the lower lobe sufficiently to produce an over-all area of 400,000 cir. mils. This it is felt will facilitate interchangeability so far as hangers and other hardware adaptation are concerned. This will be taken up with the Joint Committee on Trolley Wire Specifications.



Nominal Area, cir. mils.. 400,000
Actual Area, sq. in....................3120
Calculated Weight, lb. per 1000 ft. ...1200

Dimensions to be used by the Die Maker

A	0.232"	±0.005
B	0.203"	±0.005
C	0.310"	±0.005
D	0.700"	±0.016
E	0.423"	±0.008
F	0.080"	±0.005
G	0.015"	+0.010 -0.005
H	0.015"	+0.010 -0.005

FIG. 2.—Figure 9 Deep-Section Grooved Trolley Wire of 400,000 Cir. Mils.

RECOMMENDATIONS AFFECTING STANDARDS

I. Proposed Revision of Tentative Standard:

Tentative Specifications for Hard-Drawn Copper Alloy Wires for Electrical Conductors (*B 105 – 36 T*).[2]—Certain comments have been submitted on these tentative specifications which indicate that misunderstanding and confusion may occur relative to joints in this wire. In submitting these specifications last year, it was the committee's intention that no undrawn joints would be allowed, and also that methods other than brazing would be acceptable in making these joints. In order to obviate possible misunderstandings, the committee recommends that these tentative specifications be revised as indicated below, and continued as tentative for another year:

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[1] 1936 Book of A.S.T.M. Standards, Part I, pp. 649, 644.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 760 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 339.

New Section.—Add a new Section 6 to read as follows, renumbering the subsequent sections accordingly:

6. *Joints.*—Welds and brazes may only be made in rods or in wires prior to final drawing.

Section 6.—Change Paragraph (*c*) of the present Section 6 to read as follows by the addition of the italicized words and the omission of those in brackets:

(*c*) Tests upon a section of wire containing a drawn [braze] *joint* shall show at least 95 per cent of the tensile strength of the [unbrazed] *unjointed* wire. Elongation tests are not to be made upon test sections including [brazes] *joints.*

Section 10.—Omit Paragraph (*b*) of the present Section 10 which reads as follows:

(*b*) Necessary brazes in hard-drawn wire shall be made in accordance with the best commercial practice.

II. Proposed Revisions of Standards:

Standard Specifications for Bronze Trolley Wire (B 9 – 36)[1] *and Standard Specifications for Copper Trolley Wire (B 47 – 36).*[2]—Figure 1 of these standard specifications shows grooved trolley wire sections which are identical in dimensions as applying to both of these specifications. Experience in manufacturing these sections has shown that the tolerances indicated for the width of the bottom lobes of these sections may not practically be met, and the committee recommends that these tolerances be changed as follows, the dimensions in Fig. 1 being changed to correspond:

WIRE SIZE	PRESENT VALUE	REVISED VALUE
133,200 (00)	±0.007	+0.006 −0.012
168,100 (000)	±0.008	+0.006 −0.012
211,600 (0000)	±0.009	+0.006 −0.012
300,000	±0.011	+0.010 −0.020
350,000	±0.012	+0.010 −0.020

In view of the fact that these changes are merely a confirmation of present manufacturing practice, the committee recommends that Fig. 1 of these specifications be corrected immediately in accordance with these recommendations. It accordingly asks for the necessary nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society for immediate adoption.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 649.

[2] *Ibid.*, p. 644.

III. Tentative Standard Continued as Tentative:

Tentative Specifications for Hard-Drawn Copper Transmission Cable (B 87 - 32 T).—For several years the matter of revision of these specifications has been under consideration by Subcommittee I on Electric Transmission Wire and Cable Specifications (G. E. Dean, chairman). While some matters under discussion, such as the claimed advantage of a soft core in a hard-drawn cable, have been settled, there still exists a very decided divergence of opinion as to the desirability of the methods of inspecting and testing for tensile strength and elongation at present included in these tentative specifications. Also, it is reported that relatively small quantities of cable manufactured in accordance with these specifications have been purchased. The committee, therefore, recommends that the status of these tentative specifications be not changed at the present time, and that they be retained for another year in their present form so that additional experience may be accumulated.

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 26 members; 25 members returned their ballots, the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. Proposed Revision of Tentative Standard			
Specifications for Hard-Drawn Copper Alloy Wires for Electrical Conductors (B 105 - 36 T).....	23	0	2
II. Proposed Revisions of Standards			
Specifications for Bronze Trolley Wire (B 9 - 36), immediate adoption..........................	18	0	7
Specifications for Copper Trolley Wire (B 47 - 36), immediate adoption..........................	18	0	7

This report has been submitted to letter ballot of the committee which consists of 26 members; 24 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. A. Capp,
Chairman.

J. H. Foote,
Secretary.

REPORT OF COMMITTEE B-2

ON

NON-FERROUS METALS AND ALLOYS

Committee B-2 on Non-Ferrous Metals and Alloys, since the presentation to the Society of its report at the annual meeting in 1936, has held one meeting, in New York City on April 6, 1937.

It is with regret that the committee records the death on December 16, 1936, of Dr. William Campbell. Doctor Campbell served as chairman of Committee B-2 for 25 years, since its organization in 1909, and in 1934 he was elected to the office of honorary chairman. In Doctor Campbell's death the committee has lost a faithful member, one who rendered meritorious service to the committee and who constantly had its welfare at heart. Though his many friends and associates will sorely miss his presence, his efforts in the general advancement of technical knowledge in the field of metallurgy will be a continual source of inspiration. An engrossed memorial has been prepared, signed by the members of the Advisory Committee and forwarded to his bereaved mother.

At the present time the committee membership totals 100, of whom 47 are classified as producers, 33 as consumers, and 20 as general interest members.

A special committee under the chairmanship of Mr. T. A. Wright has completed the preparation of Regulations Governing Committee B-2 which will supplement the general Society regulations for standing committees. After being approved, with minor corrections, by the Advisory Committee, these committee regulations were submitted to the letter ballot of the committee; 67 members returned their ballots, all of whom have voted affirmatively.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Refined Copper (H. C. Jennison, chairman) has considered the desirability of preparing specifications for cathode copper, but it appears that at the present time it is unnecessary and unadvisable to proceed on this subject.

Subcommittee II on Refined Lead, Tin, Antimony, and Bismuth (T. A. Wright, chairman) has held three meetings, and has appointed three groups as follows: (*a*) For the study of copper-bearing lead for the manufacture of sheath for electrical cables; (*b*) To collect information on refined tin, the forms in which it is available, its properties, and, in conjunction with Committee E-3, methods of sampling and analysis; and (*c*) A similar survey of refined antimony.

Subcommittee III on White Metal Alloys (G. H. Clamer, chairman) held one meeting during the year at which the Standard Specifications for White Metal Bearing Alloys (Known Commercially as "Babbitt Metal") (B 23 – 26) were critically examined, and the specifications were approved in their present form. A canvass was made as to the desirability of formulating specifications for type metal, but unfortunately, there is a lack of interest in this subject on the part of the consumers. This subcommittee plans to give consideration to specifications for nickel brasses for casting purposes, containing up to 30 or 35 per cent nickel, and to reconsider the babbitt specifications in the light of quite recent changes in the manufacture and use of bearings.

Subcommittee IV on Refined Zinc and Wrought Zinc (E. H. Bunce, chairman) has completed an extensive revision of the Standard Specifications for Rolled Zinc (B 69 – 29) which is being presented for publication as tentative. In its consideration of these revised specifications the subcommittee has appointed a Special Research Committee, under the chairmanship of Mr. J. R. Townsend, on Methods of Testing Rolled Zinc. The Special Committee held its first meeting in Chicago in March during the spring group meetings of A.S.T.M. committees, at which time a coöperative program of work was planned.

Subcommittee V on Precious Metals and Alloys (R. H. Leach, chairman) held one meeting at which the Standard Specifications for Silver Solders (B 73 – 29) were critically reviewed and approved without amendment.

Subcommittee VI on Coated Metals (W. G. Schneider, chairman) has appointed a group to investigate the properties and need of specifications for copper wire coated with lead-tin alloy, a product used throughout the electrical industry.

Subcommittee VII on Refined-Nickel and High-Nickel Alloys, Cast and Wrought (C. E. Margerum, chairman) at its organization meeting appointed a group which has drafted proposed specifications for monel metal in sheet form, suitable for unfired pressure vessels, and this is now under consideration by the entire subcommittee. Another group is studying nickel and the high-nickel alloys in rods, bars, and sheets for general engineering construction.

Subcommittee VIII on Miscellaneous Refined Metals and Alloys (E. E. Schumacher, chairman) has held two meetings during the year. A group is engaged in a critical examination of the specifications under the jurisdiction of the subcommittee, all of which are nearly ten years old, and revisions in each are under discussion.

RECOMMENDATIONS AFFECTING STANDARDS

I. Proposed Revision of Standard.—In view of the fact that the Standard Specifications for Rolled Zinc (B 69 – 29)[1] have not proved very useful and do not conform to present trade practice, the committee recommends that they be revised as proposed by Subcommittee IV on Refined Zinc and Wrought Zinc (E. H. Bunce, chairman). The proposed revision is in the form of new tentative specifications, as appended hereto,[2] which are intended to replace, when adopted, the present Standard Specifications B 69 – 29.

II. Adoption of Tentative Revision of Standard as Standard.—The committee recommends that the tentative revision[3] proposed in 1935 in the Standard Specifications for Slab Zinc (Spelter) (B 6 – 33)[4] be approved for reference to letter ballot of the Society for adoption as standard. This revision will make provision for zinc of extreme purity designated as (1*a*) Special High Grade, and will bring the specifications into conformity with present-day practice.

These recommendations have been submitted to letter ballot of the committee which consists of 100 members; 65 members returned their ballots, the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. PROPOSED REVISION OF STANDARD			
Specifications for Rolled Zinc (B 69 – 29)	45	0	20
II. ADOPTION OF TENTATIVE REVISION OF STANDARD AS STANDARD			
Specifications for Slab Zinc (Spelter) (B 6 – 33)	50	0	15

This report has been submitted to letter ballot of the committee which consists of 100 members; 65 members returned their ballots, of whom 63 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

R. F. MEHL,
Chairman.

E. E. THUM,
Secretary.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 705.

[2] See p. 698.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1196 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 1334.

[4] 1936 Book of A.S.T.M. Standards, Part I, p. 714.

REPORT OF COMMITTEE B-3
ON
CORROSION OF NON-FERROUS METALS AND ALLOYS

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held one meeting during the past year, in Atlantic City, N. J., on June 30, 1936.

Subcommittees charged with the responsibility of conducting accelerated laboratory tests and the preparation of specifications covering laboratory test methods have been relatively inactive during the year. It is hoped, however, that the improvement in industry, and ideas and opinions which are bound to be forthcoming from a discussion of the papers presented at the Symposium on Corrosion Testing Procedure, held at the Regional Meeting of the Society on March 2, 1937, in Chicago, Ill., will soon make it possible for those groups to resume active laboratory work.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Spray Test (E. H. Dix, Jr., chairman).—This subcommittee has carried on its work during the year by correspondence. The principal subjects under discussion have been the effects of temperature on the results of salt spray tests and the proposal to establish a standard method for salt spray testing. Pending the holding of the Symposium on Corrosion Testing Procedure, the subcommittee felt that nothing very definite could be accomplished on either subject. A paper on "Salt Spray Testing," by E. H. Dix, Jr., and J. J. Bowman, was presented as a part of this symposium and it is hoped that this paper and the resulting discussion will permit the writing in the near future of a recommended method for salt spray testing.

Subcommittee VI on Atmospheric Corrosion (W. H. Finkeldey, chairman).—This subcommittee has submitted to its members a letter ballot on whether the next series of tests of the specimens exposed in the atmospheric corrosion tests on non-ferrous metals and alloys should be made in 1937, 1939, or 1940. The results showed that a large majority of the subcommittee members favored making these tests in 1937. In accordance with this decision, tension specimens will be removed from the test racks at all locations and distributed to the respective suppliers of the materials for testing. Plate specimens will also be removed at the same time and forwarded to the National Bureau of Standards for determinations of the changes in weight and for inspection. The removal of specimens will be started the latter part of July, 1937. It is hoped that the tension tests will be completed and the changes in weight determined before the first of September, so that in accordance with the usual custom, a meeting of the subcommittee can be held in Washington, D. C., the latter part of September to consider the test data and inspect the plate specimens.

Attention is called to a correction that should be noted in the 1935 report of Subcommittee VI. In the last paragraph under "Tension Tests," that part of the paragraph explaining the use of the symbols X and s in Table II should be deleted, since changes made in this table after the report was in preprint form made the use of these symbols unnecessary. The paragraph in question, which appears at the top of page 147 of the 1935 *Proceedings*, Part I, should be changed to read as follows by the omission of the words in brackets:

> In lieu of such written comment, Table II has been prepared in which the amount of corrosion shown by the materials is classified as "negligible" (left blank in the tables) if the differences between the "percentage change" figures and their respective "maximum percentage difference due to chance" values are less than 1 per cent [and classified as "small" (indicated by s) if the differences are from 1 to 3 per cent and as "significant" (indicated by X) if the differences are 3 per cent or greater].

Subcommittee VIII on Galvanic and Electrolytic Corrosion (C. L. Hippensteel, chairman).—This subcommittee plans during the coming year to start the stainless steel - non-ferrous metal couple tests, and also the salt-water immersion couple tests.

This report has been submitted to letter ballot of the committee which consists of 52 members; 44 members returned their ballots, of whom 42 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

T. S. FULLER,
Chairman.

SAM TOUR,
Secretary.

REPORT OF COMMITTEE B-4

ON

ELECTRICAL-HEATING, ELECTRICAL-RESISTANCE AND ELECTRIC-FURNACE ALLOYS

Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys held two meetings during the year, in Cleveland, Ohio, on September 28 and 29, 1936, and in New York City, on February 18 and 19, 1937, in addition to the meeting held during the annual meeting of the Society.

The committee consists of 42 members, of whom 18 are classified as producers, 18 as consumers and 6 as general interests.

RECOMMENDATION AFFECTING STANDARD

Proposed Revision of Tentative Standard.—The committee recommends that the Tentative Method of Test for Flexivity of Thermoflex (Thermostatic Metals) (B 106 – 36 T)[1] be revised as follows and continued as tentative:

Section 2.—Change Paragraph (*e*) covering the definition of flexivity from its present form to read as follows:

(*e*) *Flexivity, F.*—The flexivity, *F*, is the change of curvature of the longitudinal center line of the specimen per unit temperature change for unit thickness, and is given by the formula:

$$F = \frac{\left(\frac{1}{R_1} - \frac{1}{R_0}\right)t}{T_1 - T_0} \qquad (1)$$

whence, referring to Fig. 1:

$$F = \frac{8t}{T_1 - T_0}\left(\frac{D_1}{Q^2 + 4D_1t + 4D_1^2} - \frac{D_0}{Q^2 + 4D_0t + 4D_0^2}\right) \qquad (2)$$

where

R = the radius of curvature, in inches,
t = the thickness, in inches,
D = the deflection, in inches, as measured with reference to the plane including the line AB and parallel to the knife edges (see Fig. 1),
Q = the distance between knife edges, in inches,
T = the temperature, in degrees Fahrenheit,
$T_1 - T_0$ = the change in temperature in degrees Fahrenheit,
D_0 = D corresponding to T_0, and
D_1 = D corresponding to T_1.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 765 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 360.

Equation (2) is an exact expression which takes into account initial curvature If the specimen is accurately straight at T_0, then $\frac{1}{R_0}$ in Eq. (1) is zero, D_0 is zero, and Eq. (2) reduces to:

$$F = \frac{8t}{T_1 - T_0}\left(\frac{D_1}{Q^2 + 4D_1t + 4D_1^2}\right) \qquad \text{(3)}$$

The recommendation appearing in this report has been submitted to letter ballot of the committee, which consists of 42 members; 31 members returned their ballots, of whom 26 have voted affirmatively, none negatively, and 5 marked their ballots "not voting."

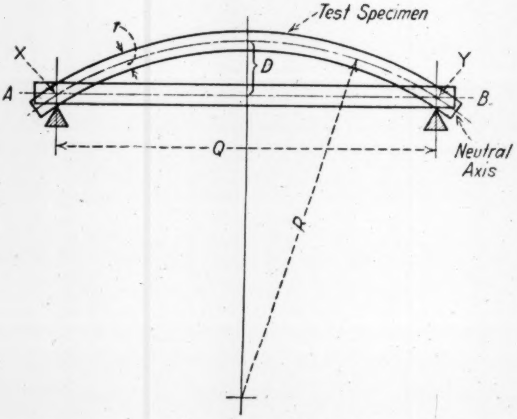

FIG. 1.—Test for Flexivity of Thermoflex.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Life Tests (J. W. Harsch, chairman).—In connection with the Standard Accelerated Life Test for Metallic Materials for Electrical Heating (B 76 – 36), the interlaboratory tests of the glass slide in the life test equipment for protection against temperature variations have nearly been completed.

The high-temperature accelerated life test for electric-furnace resistor alloys is being given a trial in four laboratories with different methods of suspension of the specimen.

Subcommittee II on Electrical Tests (H. L. Curtis, chairman).—A method of test for temperature coefficient of manganin is in course of preparation, and further work is being done on the heat treatment of the specimen.

Subcommittee IV on Mechanical Tests (P. H Brace, chairman).—The three types of bend testing machines in the Proposed Method for the Bend Testing of Wire,[1] which was published as information with the report of the committee last year, are being studied together with a torsion test. Comparative tests have been made on three sizes of nickel wire with three conditions of cold work and three conditions of anneal. These samples were chosen to indicate the ability of the various types of equipment to distinguish small differences in physical properties of the wire. The tests have been made and are being analyzed preparatory to formulating the method for publication as tentative.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 162 (1936).

Subcommittee V on Wrought and Cast Alloys for High-Temperature Use (C. L. Clark, chairman).—This subcommittee has completed proposed appendices to the Tentative Methods of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 34 T) and for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 – 35 T) which cover the preparation of cast tension specimens of heat resistant metals (chromium-nickel, chromium-nickel-iron, and chromium-iron). They include specimens with shanks sufficiently long to remove threaded connections from the heated zone of the furnace, and describe a casting procedure which has been found satisfactory for producing sound castings. The appendices have been submitted for approval to the Joint Research Committee on Effect of Temperature on the Properties of Metals.

The equipment for the high-temperature bend test has been completed and tests are being made.

A warpage test specimen in the form of an eccentric tube is being studied, with specimens of both cast and wrought materials, to determine the relative tendency of alloys to warp at elevated temperatures. The specimens are quenched in water or in oil to expedite the test.

Subcommittee VII on Thermostatic Metals (P. H. Brace, chairman).—The Tentative Method of Test for Flexivity of Thermoflex (Thermostatic Metals) (B 106 – 36 T) is being studied to eliminate possible causes of variation and to determine the effect of width of specimen. The definition for flexivity is being modified, as indicated earlier in this report.

Methods of evaluating stiffness, elastic strength and permissible range of operating temperature also are being studied.

Subcommittee VIII on Metallic Materials for Radio Tubes and Incandescent Lamps (A. L. Chapman, chairman).—The Radio Manufacturers Association has requested the Society to prepare methods of test for metallic materials used in radio tubes and this work, with the related work on metals in incandescent lamps, has been assigned to Committee B-4. A new subcommittee, composed of 20 members, has been organized with A. L. Chapman as chairman and F. E. Bash as secretary. One meeting of the subcommittee has been held at which the proposed work was divided among the following five sections: on Strip, Wire, Tubing, Coated Material, and Powdered Materials and Liquids.

Effect of Controlled Atmospheres Upon Electric Furnace Resistors and Structures.—The electric furnace industry has had some difficulty due to the deterioration of resistors subjected to controlled atmospheres in electric furnaces. In response to their request, Committee B-4 is arranging for the organization of a new subcommittee to undertake the development of a suitable method of test to determine the durability of resistor alloys exposed to controlled atmospheres at high temperatures in electric furnaces.

This report has been submitted to letter ballot of the committee which consists of 42 members; 31 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

DEAN HARVEY,
Chairman.

F. E. BASH,
Secretary.

DISCUSSION

The Chairman (*Mr. W. R. Webster*[1]).—Mention is made in the report of Committee B-4 of the effect of the circulation of atmosphere on resistor elements. In view of the extent to which circulated atmospheres in furnaces are coming into use, has anything quantitatively been done on that, because the circulation is pretty vigorous in some of the furnaces now on the market?

Mr. Dean Harvey.[2]—What we are trying to do is to establish for our own investigation one standard atmosphere, and a method of test to determine the corrosive effect of that atmosphere upon the resistor element, and make tests in different laboratories to see whether we can get good checks. When we can get a good check between laboratories, we desire to submit a standard method of test which can be used by any company with any atmosphere which they desire to investigate.

Mr. Webster.—What I had in mind is this: There are two materials which in a perfectly stationary atmosphere have certain relations to each other—one better than the other. That might be reversed in the presence of a vigorous circulation. I should think that was a very important matter to investigate.

Mr. Harvey.—Yes, it is, but we have not gone quite that far yet.

Mr. Webster.—It is a very active committee. I congratulate you, sir.

Mr. Harvey.—This work will require careful attention. We have limited the subcommittee to nine members, all of whom are connected with laboratories which are in a position to work on this subject.

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[1] Chairman of the Board, Bridgeport Brass Co., Bridgeport, Conn.

[2] Materials Engineer, Central Material and Process Engineering Dept., Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

REPORT OF COMMITTEE B-5
ON
COPPER AND COPPER ALLOYS, CAST AND WROUGHT

Committee B-5 on Copper and Copper Alloys, Cast and Wrought, held two meetings during the year, at Atlantic City, N. J., on July 2, 1936, and at New York City, on April 6, 1937.

Subsequent to its meeting last June, Committee B-5 presented to the Society, through Committee E-10 on Standards, revisions of the following four specifications:

Tentative Specifications for:

Copper-Silicon Alloy Plates and Sheets (B 96 – 36 T),
Sheet Copper-Silicon Alloy (B 97 – 36 T),
Copper-Silicon Alloy Rods, Bars and Shapes (B 98 – 36 T), and
Copper-Silicon Alloy Wire for General Purposes (B 99 – 36 T).

The revisions recommended in these specifications were accepted[1] by Committee E-10 on August 26, 1936, and the specifications in their revised form appear in the 1936 *Proceedings*.[2]

RECOMMENDATIONS AFFECTING STANDARDS

I. Proposed Tentative Standard.—The committee recommends for publication as tentative the proposed Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock, appended hereto.[3] These specifications were prepared by Subcommittee I on Wrought Metals and Alloys (R. A. Wilkins, chairman) for the purpose of combining in a single standard the detailed specification requirements for the various alloys manufactured in the form of seamless tubes for use in surface condensers, evaporators and heat exchangers, including admiralty metal, Muntz metal, and copper-nickel alloy. The existing standard and tentative specifications covering these alloys are, therefore, being recommended for withdrawal.

II. Revision of Tentative Standard.—The committee recommends that the Tentative Specifications for Copper-Silicon Alloy Wire for General Purposes (B 99 – 36 T)[4] be revised as indicated below and continued as tentative:

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[1] In submitting these revised tentative specifications to Committee E-10 on Standards, Committee B-5 reported the following results of the letter ballot vote of a total of 47 ballots returned from a committee membership of 64: Specifications B 96, affirmative 35, negative 0, ballots marked "not voting" 12; Specifications B 97, affirmative 34, negative 0, ballots marked "not voting" 13; Specifications B 98, affirmative 36, negative 0, ballots marked "not voting" 11; Specifications B 99, affirmative 32, negative 1, ballots marked "not voting" 14.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 744, 751, 747 and 756 (1936); also 1936 Book of A.S.T.M. Tentative Standards, pp. 319, 326, 322 and 331.

[3] See p. 684.

[4] ***Proceedings***, **Am. Soc. Testing Mats., Vol. 36, Part I, p. 756 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 331.**

Section 3.—In the table of chemical composition change the minimum copper content for Type B alloy from "97.00" per cent to read "95.00" per cent.

In the list of specified elements add the following requirements for aluminum, to appear after the requirements for tin:

	TYPE A	TYPE B	TYPE C	TYPE D
Aluminum, max., per cent............	....	2.00	2.00	....

III. Withdrawal of Standards and Tentative Standard.—It is recommended that the following three specifications, which are being replaced by the proposed Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock, as mentioned earlier in this report, be withdrawn:

Standard Specifications for:

Seamless Admiralty Condenser Tubes and Ferrule Stock (B 44 – 33), and
Seamless Muntz Metal Condenser Tubes and Ferrule Stock (B 56 – 33).

Tentative Specifications for Seamless Copper-Nickel Alloy Condenser Tubes and Ferrule Stock (B 104 – 36 T).

The committee also recommends the withdrawal of the Standard Specifications for Seamless 70–30 Brass Condenser Tubes and Ferrule Stock (B 56 – 33) as the alloy covered by these specifications has outlived its usefulness.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 66 members; 50 members returned their ballots, the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. PROPOSED TENTATIVE STANDARD			
Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock........	33	0	17
II. REVISION OF TENTATIVE STANDARD			
Specifications for Copper-Silicon Alloy Wire for General Purposes (B 99 – 36 T)................	31	0	19
III. WITHDRAWAL OF STANDARDS AND TENTATIVE STANDARD			
Standard Specifications for Seamless Admiralty Condenser Tubes and Ferrule Stock (B 44 – 33)..	33	0	17
Standard Specifications for Seamless 70–30 Brass Condenser Tubes and Ferrule Stock (B 55 – 33)..	32	0	18
Standard Specifications for Seamless Muntz Metal Condenser Tubes and Ferrule Stock (B 56 – 33).	32	0	18
Tentative Specifications for Seamless Copper-Nickel Alloy Condenser Tubes and Ferrule Stock (B 104 – 36 T)..	33	0	17

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Wrought Metals and Alloys (R. A. Wilkins, chairman).—This subcommittee has nearly completed its work on the preparation of new specifications for sheet and strip brass, considerably enlarging the scope of the present Standard Specifications for Sheet High Brass (B 36 – 33) and including carefully considered precision requirements for the chemical composition of this material. The subcommittee also has in preparation new specifications for rolled non-ferrous bearing plates for bridges and other structural uses.

Subcommittee II on Cast Metals and Alloys (G. H. Clamer, chairman).—Considerations being given by this subcommittee to adjustments in the specifications covering the various cast alloys to correspond with the related ingot metal specifications have not yet progressed far enough to make possible a final disposition of the Tentative Specifications for Sand Castings of the Alloy: Copper 80 per cent; Tin 10 per cent; Lead 10 per cent (B 74 – 32 T), which must be considered in relation to other alloys. It is expected that recommendations arising from the general program will be made during the coming year.

At the spring meeting of Committee B-5 the desire of the chairman and secretary to retire after nearly ten years of service was recognized by the appointment of a committee under the chairmanship of G. H. Clamer to report at the June meeting on nominations for new officers. An election of officers held at the meeting of the committee during the annual meeting resulted in the selection by unanimous approval of Mr. C. H. Greenall as Chairman and Mr. C. H. Davis as Secretary to serve until 1938.

This report has been submitted to letter ballot of the committee which consists of 66 members; 50 members returned their ballots, of whom 48 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

C. H. MATHEWSON,
Chairman.

D. K. CRAMPTON,
Secretary.

REPORT OF COMMITTEE B-6
ON
DIE-CAST METALS AND ALLOYS

Committee B-6 on Die-Cast Metals and Alloys has held two meetings during the year, one at Atlantic City, N. J., on July 1, 1936, and one at Chicago, Ill., on March 2, 1937, with an attendance at the two meetings, of 24 and 38, respectively. The subcommittees have also held meetings at various times during the year.

The following additions and changes in membership have been approved:

F. A. Porter, Precision Castings Co. Inc.;

J. M. G. Fullman, National Electric Products Corp.;

F. J. DeWitt, Jr., Parker Rust-Proof Co.;

J. B. Johnson, Air Corps, U. S. A., resigned from the committee;

J. C. Jones, British Aluminum Co., replacing A. J. Field, the former representative of the British Aluminum Co., who resigned from the committee to return to England;

Zay Jeffries now represents the Incandescent Lamp Division of the General Electric Co. at Nela Park, Cleveland, Ohio, instead of the Aluminum Company of America; and

R. G. Roshong has resigned and was replaced by E. C. Roglin, who will represent the Hoover Co.

At the present time the committee consists of 62 members, of whom 24 are classified as producers, 22 as consumers, and 16 as general interest members.

The present officers of the committee, J. R. Townsend, chairman, W. H. Graves, vice-chairman, and P. V. Faragher, secretary, were unanimously re-elected for another two-year term, ending in June, 1938.

Committee B-6 has advised Committee A-1 on Steel that the latter's Subcommittee XXIII on Die Steels as applied to die castings may be disbanded until such time as Committee B-6 feels that sufficient information has been collected on die-casting practices to recommend to Committee A-1 that standardization of the die steels in common use will be in order.

A paper on "Brass Die Castings" has been prepared by J. C. Fox and appears as Appendix I to this report.

RECOMMENDATIONS AFFECTING STANDARDS

Proposed Revisions of Tentative Standards.—The committee recommends that the Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 – 33 T)[1] be revised, as appended hereto,[2] and continued as tentative.

The committee also recommends that the Tentative Specifications for Magnesium-Base Alloy Die Castings (B 94 – 34 T)[3] be revised as follows and continued as tentative:

Section 1.—In the second sentence of this section change Alloy No. 2 to read Alloy No. 12.

Section 3.—The present composition known as Alloy No. 2 is to be replaced by Alloy No. 12. Alloy No. 12 differs from Alloy No. 2 in that a maximum of 1 per cent of silicon is recommended while the copper and nickel contents are limited to 0.05 and 0.03 per cent, maximum, respectively. It is accordingly recommended that the table of chemical composition be changed to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

Magnesium, min. per cent	[88.0] *remainder*
Aluminum, per cent	9.0 to 11.0
Silicon, max., per cent	*1.0*
Manganese, min., per cent	0.10
Copper, max., per cent	[0.10] *0.05*
Nickel, max., per cent	[0.05] *0.03*
Other Ingredients (zinc, cadmium, tin):	
Individually, max., per cent	*0.3*
Total, max., per cent	*0.5*

Explanatory Notes.—In the first sentence of Notes 1 and 2 change Alloy No. 2 to read Alloy No. 12.

Change the fourth and fifth paragraphs of Note 2 to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> The copper and nickel contents are set at [0.10 and] 0.05 *and 0.03* per cent, *maximum*, respectively, inasmuch as larger percentages of these elements decrease the corrosion resistance of the alloy.
>
> The present phraseology for expressing the composition of these magnesium-base die castings permits the allowable presence of appreciable amounts of metals such as [silicon] zinc, cadmium, and tin that are often present in other magnesium-base alloys and which, in small quantities, have no detrimental effects on magnesium-base alloy die castings. Such metals, therefore, should not be classed as impurities.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 62 members; 53 members returned their ballots, the result being as follows:

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 641 (1933); also 1936 Book of A.S.T.M. Tentative Standards, p. 348.

[2] See p. 705.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 703 (1934); also 1936 Book of A.S.T.M. Tentative Standards, p. 353.

Items	Affirm-ative	Neg-ative	Ballots Marked "Not Voting"
PROPOSED REVISIONS OF TENTATIVE STANDARDS			
Specifications for Aluminum-Base Alloy Die Castings (B 85 - 33 T)	51	0	2
Specifications for Magnesium-Base Alloy Die Castings (B 94 - 34 T)	51	0	2

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum-Base Die-Casting Alloys (E. E. Thum, chairman).—This subcommittee has revised the Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 – 33 T) by the addition to the table in the appendix to the specifications of test values showing the physical properties for Alloys Nos. XI and XII that may be expected of tension and impact specimens when made in a die. This revision of the specifications has been called to the attention of Committee B-7 on Light Metals and Alloys, Cast and Wrought, so that that committee will include ingots of the proper composition in the specifications for aluminum-alloy ingots for die castings which it has in preparation.

Subcommittee II on Zinc-Base Die-Casting Alloys (C. E. Heussner, chairman).—Work is under way in this subcommittee for the inclusion of Alloy No. XXV in the Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 – 34 T).

Subcommittee III on Tin- and Lead-Base Die-Casting Alloys (G. O. Hiers, chairman).—This subcommittee has formulated plans for the study of five tin-base and lead-base alloys to determine the following properties: Tensile strength, creep, impact, and hardness. The composition of these alloys is to be determined by chemical and spectrographic analyses. Several companies have agreed to supply the necessary test specimens to carry out this investigation.

Subcommittee IV on Physical Tests of Die Castings (R. L. Templin, chairman).—At a meeting of this subcommittee in March, 1937, the general subject of die castings was reviewed and new methods of testing were discussed. It was agreed that the round tension specimen should be standard since it gives more consistent results than the flat specimen. It was reported that some work is in progress in the use of a notched impact specimen in which the notch is cast into the bar. Various other tests are in progress, such as creep testing and endurance tests at elevated temperatures.

Consideration has also been given to the physical tests to be made on the tin-base and lead-base alloys. Various testing laboratories will be requested to give information regarding the standard rate of pulling the test specimens, type of machine used, grips and all other data bearing on the subject. A report on the correlation of testing practices will be presented by this subcommittee sometime in the future.

Subcommittee V on Exposure and Corrosion Tests (W. M. Peirce, chairman).—In line with the exposure and corrosion test program of Subcommittee V, three zinc-base alloys and four magnesium-base alloys will be placed on the exposure racks. The zinc-base alloys will be prepared by the Apex Smelting Co., National Cash Register Co., the New Jersey Zinc Co., Bunker Hill Zinc Producers, Stewart Die-Casting Corp., and the Superior Die Casting Co. The magnesium-base alloy specimens will be produced by the Hoover Co., Dow Chemical Co., and the American Magnesium Corp.

It was agreed that Alloy No. 12 of the Tentative Specifications for Magnesium-Base Alloy Ingot for Remelting (B 93 – 36 T) will be included in the exposure tests under the designation of Alloy No. 312 and an alloy of the composition of No. 7 under the designation 307. Two additional alloys designated 313 and 314 will also be included, the former having a nominal composition of 9 per cent aluminum and 0.6 per cent zinc, the latter differing from 307 by the addition of 2.5 per cent zinc. The New Jersey Zinc Co. will analyze the zinc-base specimens, and the Dow Chemical Co., the magnesium specimens. All of the zinc alloys will be die cast in accordance with the procedure outlined in the report by G. L. Werley on "A Study of Die Design Changes for the Improvement of the Soundness and Uniformity of Test Bars," which appears as Appendix II to this report. The manufacturers of the magnesium-base alloy specimens will arrive at a common agreement as to the standard conditions of casting in order that variables will be reduced to a minimum.

Subcommittee VI on Finishing Properties of Die Castings (J. C. Fox, chairman).—The work on metal finishes being carried out by the Joint Committee on Exposure Tests of Plating on the Non-Ferrous Metals, made up of representatives of the American Electro-Platers' Society, the National Bureau of Standards and the A.S.T.M., is well under way.

Subcommittee VII on Magnesium-Base Die-Casting Alloys (J. A. Gann, chairman).—This subcommittee at its meeting in March, 1936, approved minor revisions of the Tentative Specifications for Magnesium-Base Alloy Die Castings (B 94 – 34 T) which were subsequently approved by Committee B-6 but inadvertently omitted from the 1936 annual report and hence were not submitted to the Society last year. To correct this oversight the changes proposed in these specifications are now being presented, as mentioned earlier in this report.

This report has been submitted to letter ballot of the committee which consists of 62 members; 53 members returned their ballots, of whom 51 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

P. V. FARAGHER,
Secretary.

J. R. TOWNSEND,
Chairman.

APPENDIX I

BRASS DIE CASTINGS

By J. C. Fox[1]

The die casting of copper base alloys has had the attention of die casting engineers for many years. As far back as 1914 attempts were made to cast brass and other copper alloys with the casting machines and methods then available. It was found that these methods and machines, which were designed for zinc and aluminum die castings, could not be successfully used for the casting of brass.

One early experiment in brass casting consisted of enclosing in the die a ladle of molten brass, tipping the ladle from the outside, and allowing the molten brass to flow into the die cavity with the aid of compressed air. Brass die castings were actually produced by this method, but the process proved too costly and impractical. The method was not only slow but it was necessary to keep the metal extremely hot in order to maintain a fluid state, during the operation of closing the die and completing the casting cycle. This high temperature shortened, considerably, the life of the die.

Another process consisted of a machine which embodied the principle of charging with molten brass, an asbestos cup or shell set in a chamber above the die opening. The use of the asbestos cup prevented rapid chilling of the metal until pressure could be applied by means of a mechanical ram which forced the molten alloy into the die. This process proved to be too cumbersome, complicated and costly, and the resulting castings were excessively porous and unsound, due to the hygroscopic nature of the asbestos paper. In this process also the die life was extremely limited due to the high temperature of the alloys used in casting.

In all the early attempts to produce satisfactory brass die castings on an economical basis, the alloy was cast in the fully liquid state at temperatures considerably above the melting points. At these temperatures, difficulties were encountered due to the rapid failure of dies which were fabricated from steels not adaptable to this type of work. The ready volatilization of zinc from the brass alloys tended to coat the die surfaces heavily with zinc oxide which materially affected surface finish on the casting. The high shrinkage of the alloy at these temperatures also gave serious trouble.

It was not until a few years ago that a new process and a new type of casting machine were developed which made the pressure die casting of brass and kindred alloys a commercial reality. In the late twenties, Polak of Czechoslavakia introduced a high-pressure machine and method for casting brass. By using high pressures, it was found practical to force brass into die cavities at temperatures considerably lower than the temperatures previously used for casting the alloy. In this process, the metal is held in a furnace as a unit apart from the casting machine. The metal at a temperature close to the melting point is ladled into a *cold* chamber of the casting machine where it partially solidifies before the application of pressure forces it into the die.

Since this development, there have been a number of modifications of the Polak machine, all of which, while varying in some degree, utilize the same mechanical principle of casting and employ high pressures. Pack[2] has ably described the Polak,

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[1] Chief Chemist and Metallurgist, Doehler Die Casting Co., Toledo, Ohio.

[2] C. Pack, "Press Castings," *Metals and Alloys*, March, 1932. C. Pack, "Press Casting Brass and Other Copper Alloys," *Metal Industry*, Vol. 32, Nos. 5 and 6, May and June, 1934, pp. 155–157, and 199–201.

Pack, Doehler and other machines, while Sieg[1] has given the details of the Polak process.

This new process for brass alloys is now being extended to the casting of other alloys, notably magnesium and aluminum alloys. The higher pressures used have been found to produce higher physical properties, better surface finishes, and freedom from porosity in the casting.

The Casting Cycle:

In the operation of the Polak type of casting machine the die is closed, and the brass alloy in a liquid or semiliquid state, at a temperature very close to the melting point, is ladled into a cup or chamber on top of an ejector plunger. The ejector plunger at this point blocks the gate or inlet into the die. Above the cylinder is a hydraulic ram controlled by a valve which the casting operator opens immediately after the alloy is ladled into the chamber. The ram forces the ejector plunger downward, opening the gate to the die, and as the ram continues downward, the metal is forced into the die under pressures ranging from 3000 to 10,000 lb. per sq. in. When the pressure is released and the ram withdrawn, the ejector plunger, through its spring action, is forced upward causing it to shear off the gate sprue and eject the solid slug of excess metal. The cycle of casting operations is then completed by opening the die and ejecting the casting.

Applications in Industry:

The salient qualities of brass die castings, high strength and corrosion resistance, make them suitable for many diversified uses. The die casting of high tensile strength alloys opens up to industry a source of engineering parts of high strength, intricacy, accuracy, stability, and economy. It is difficult to limit the potential market of materials having such a combination of properties. The variety of possible uses is far too wide to be given in this paper; however, the following is a partial list of the present and possible applications of brass die castings:

In the automotive industry—gears, transmission forks, clutch parts, hydraulic brake parts, shock absorber parts, pumps and bearings; in the electrical field—switchboard parts, contactor parts, brushholders, refrigerator parts for resistance to corrosion and to the chemical action of refrigerants; ordnance and gun parts; marine parts where strength and corrosion resistance are paramount; steam fittings and valves; trunnion bearings; oil burner parts; oil well and hydraulic pump parts; general engineering fittings; household hardware; and many others. In one application, that of a shock absorber piston more than a million brass die casting parts have been produced during the past year.

DIES

Die Steels:

Since the first production of brass die castings on a commercial scale, attention has been concentrated on obtaining the most suitable composition and method of heat treating steel to give satisfactory die life. It has become apparent that the steels and heat treatments previously used for die casting aluminum with temperatures up to 1300 F. were definitely unsuited for brass with casting temperatures up to 1700 F. It was found that a much greater difference could be expected in the performance of dies between those used for brass and aluminum than was experienced between those used for aluminum and zinc or other lower melting point alloys.

The extreme surface temperature of the die in brass die casting may rise higher than 1600 F., a temperature well beyond the transformation range of steels. The continued heating of the surfaces to this point results in reduction of strength and hardness, lowers the resistance of the steel to erosive action of the molten alloy, and generally nullifies the effects of heat treatment.

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[1] W. W. Sieg, "Brass Pressure Castings are Produced Economically," *The Iron Age*, November 30, 1933, pp. 16–19.

A suitable steel for brass die casting dies must possess very high red-hardness properties and retain hardness and other properties at high temperatures. In addition, a suitable brass die steel must possess the following characteristics:

1. High impact strength and toughness at ordinary temperatures,
2. High impact strength and toughness at elevated temperatures,
3. Resistance to cleavage cracking or cracking due to sudden changes of temperature,
4. Resistance to heat checking,
5. Resistance to solvent or erosive action of molten alloy,
6. Relative ease of heat treatment and stability in heat treatment, and
7. Relative ease of machinability.

In the course of development of the brass die casting process, many compositions of steel covering practically all available types of alloy steels have been tried under actual service conditions. These include the high-chromium (18 to 30 per cent), the high-chromium-nickel stainless and heat-resistant steels, the high-speed steels with varying content of tungsten and other elements, and a large variety of new alloy steel compositions with high content of chromium, molybdenum, cobalt, and other elements.

Although the life of dies is steadily improving, the development of special steels for the casting process is a pressing need, one which should challenge the ability and ingenuity of every steel metallurgist. In this the steel manufacturer has an open field for the development of a material which will meet all the conditions and requirements imposed by this process.

The type of steel now generally used for brass die casting dies is one of the so-called hot work type, which is used for dies for forging, swaging extrusion, and similar methods. The composition lies between the following limits:

Carbon, per cent	0.30 to 0.40
Tungsten, per cent	8.00 to 15.00
Chromium, per cent	2.75 to 3.25
Vanadium, per cent	0.25 to 1.50
Silicon, per cent	0.20 to 0.50
Manganese, per cent	0.20 to 0.50
Sulfur, per cent	under 0.02
Phosphorus, per cent	under 0.02

This steel is used only because there has been no better one available, although there are now on test some very promising new types of composition which will undoubtedly replace it.

Particular care must be taken in the heat treatment of the chromium-tungsten steel. It must be heated carefully and to a sufficiently high temperature to insure as complete a solution of the carbides as is possible, to minimize susceptibility to cleavage cracking. A carefully regulated atmosphere-controlled furnace must be used to prevent decarburization and carburization as, in die casting dies, no extra stock can be allowed for grinding after heat treatment as is the case in the manufacture of other tools. Decarburization of the surface of the die causes rapid heat checking.

The final Brinell hardness after heat treatment must be within definite limits. The shock resistance of the steel is greatly lowered when the Brinell hardness exceeds 444; heat checking is apt to take place rapidly when the hardness drops below 430.

Heat Treatment:

The steps in the method of heat treatment used for this steel are essentially as follows:

1. Pack.
2. Preheat the die blocks by slowly heating to 1550 to 1600 F.
3. Transfer to high-temperature atmosphere-controlled electric furnace and heat to 2190 to 2200 F.
4. When blocks have attained this temperature, continue heating until blocks are thoroughly heated throughout.
5. Quench in oil.
6. Draw at 1135 to 1150 F., and soak at this temperature for a time equal to 6 hr. per inch of thickness.
   Final Brinell hardness between 430 and 444.

Die Life:

With the present die steels, the life of a die may range between 10,000 and 50,000 operations before replacement becomes necessary. The die life appears to be a function of the size, weight, and wall section of the casting. Under the same conditions dies for thin-walled castings greatly outlast those for castings of heavy section. This may be accounted for by the greater amount of heat absorbed by the die from the larger amounts of metal required for heavier castings.

For small thin-walled castings a die life of 50,000 or more cycles can be expected. Some parts, particularly the gate block and sprue, require replacement oftener than the impression parts. The gate block is the first to receive the full charge of metal and consequently is always heated to higher temperatures than the more remote parts of the die. During steady casting operation, these parts reach a dull red heat and remain at this temperature during casting. This clearly indicates the necessity for a steel of high red-hardness for brass die casting dies.

Die Design:

Dies for casting brass are fabricated in a manner similar to those for casting aluminum and zinc. The brass dies are generally made up in small sections, each impression being a separate block in order to facilitate handling in heat treatment and for ease of replacement.

The methods of gating and venting for brass are similar to those used for other die-cast alloys. Because of the higher pressures used on brass, the size of the gate inlet required is much smaller than that necessary for the same shape and size of casting in zinc and aluminum.

At the present stage of development, 5 lb. is generally considered the maximum weight for brass die castings, although some larger castings have been produced. The average weight of castings is approximately 1.5 lb.

The minimum wall thickness of brass die castings can be placed at $\frac{1}{16}$ in. with an average of $\frac{3}{32}$ in. The wall stock required naturally depends upon the size, shape, design, and nature of the application. Dimensional tolerances also depend upon the size and shape of the casting. The magnitude of the dimension is also an important consideration in fixing the limits. Uniformity of wall sections and the avoidance of thick sections wherever possible are also important in the control of limits. It is well to keep the sections uniform and avoid too great a variation in adjacent sections. The walls of castings require a draft or taper of $\frac{1}{64}$ in. per inch on a side. Cores must also be given adequate draft.

Holes of minimum diameter of $\frac{3}{16}$ in. are cast. A taper of $\frac{1}{64}$ in. per inch of depth of hole is necessary. As a general rule no threads are cast. Coarse external threads may be cast in some instances, but for the most part, it is found more economical to cut threads than to cast them, since cast threads require chasing and a cut thread is usually cleaner and sharper than a cast one.

In many cases, it is possible to keep within narrower limits in casting than those given above, but this again depends upon conditions and the general practice in designing a casting is to allow as much tolerance as is possible to meet all requirements. Working within unnecessarily narrow limits adds to costs without any benefits being derived.

ALLOYS

The important factors controlling the quality of a brass die casting alloy may be listed as follows:

1. Melting point.
2. Physical properties.
3. Physical properties at elevated temperatures.
4. Fluidity.
5. Machinability.
6. Action on steel die—erosive or solvent action.
7. Corrosion resistance.
8. Cost.

As the difficulties encountered in the production of die castings multiply rapidly with increasing melting points of the alloys used, it is obviously necessary to use only the lower point alloys. The choice of the alloy must, of course, be consistent with good casting properties, suitable mechanical properties, machinability, and other necessary properties.

The 60 per cent copper, 40 per cent zinc type alloy which has the lowest melting point of the commercial *high* brasses, was the one first to be used and is still being used. In the die-cast state this alloy has good physical properties and corrosion resistance.

Aluminum bronze, tin bronzes, red brass, 85-5-5-5 and similar high-copper alloys can be readily cast by the process described but the melting points and casting temperatures are too high to be practical or economical with the die materials at present available.

Added Elements and Impurities:

The effect of added elements and impurities in brass die casting alloys is very pronounced and of such importance as to warrant discussion here.

Lead.—Lead, which is practically insoluble in copper and brass, is usually added to sand-cast and forging brass alloys up to 3.00 per cent to improve machining characteristics. In the solid alloy, it is distributed as globules in the grain boundaries forming planes of weakness, which make for ease in machining. For brass die casting alloys, however, lead in any but the smallest amount appears to be more harmful than beneficial, for the same effect of lead in sand-cast and forged parts is not found with brass die castings. Lead in brass die castings causes a reduction in strength and ductility and there is great tendency towards segregation and liquation of the lead, producing unsightly gray spots on the casting. It also contributes to hot-shortness of the alloy in casting. Therefore, when the optimum in physical properties is desired the lead content should be kept below 0.25 per cent.

Tin.—Tin increases the hardness. Over 1 per cent tin in the alloy for brass die castings results in difficulty in machining. The advantages of tin additions are increased corrosion resistance and better surface finish.

Bismuth.—The effect of bismuth is particularly harmful on the mechanical properties of brass and copper alloys because of its distribution in the grain boundaries causing brittleness. The bismuth content should be kept below 0.005 per cent.

Antimony.—The effect of anitmony on brass is similar to that of bismuth; the content should be kept below a maximum of 0.005 per cent.

Arsenic.—The presence of arsenic in the 60 per cent copper—40 per cent zinc brass alloy is highly deleterious; as little as 0.05 per cent reduces ductility greatly.

Sulfur, Tellurium, and Selenium.—Sulfur, tellurium, and selenium are regarded as harmful on the mechanical properties of brass and should be held down to the very minimum. Sulfur present as cuprous sulfide reduces ductility.

Iron.—A small amount of iron in brass considerably increases the hardness, lightens the color, and reduces corrosion resistance. In brass die castings, it should be kept below 0.25 per cent.

Manganese.—The apparent effect of manganese is that it hardens the brass, increases tensile strength, and decreases elongation. The alloy known as manganese bronze, which is essentially a brass alloy containing small percentages of manganese, has been die cast with satisfactory results.

TABLE I.—MELTING POINTS OF COPPER—ZINC ALLOYS.

Alloy Composition,		Temperature,	
Copper, per cent	Zinc, per cent	Deg. Cent.	Deg. Fahr.
100	0	1082	1980
96	4	1075	1967
86	14	1032	1890
80	20	1008	1846
76	24	980	1796
72	28	958	1756
71	29	952	1746
66.4	33.6	918	1684
63	37	908	1666
60	40	904	1655
50	50	880	1616
48	52	870	1598
41	59	840	1544
35	65	816	1501
33	67	803	1477
29	71	786	1467
24	76	740	1364
20	80	705	1301
15	85	657	1212
11	89	588	1090
8	92	547	1017
3.5	96.5	465	869
2	98	425	797
0	100	419	786

Magnesium.—Because of the great affinity magnesium has for oxygen, it is considered a good deoxidizer for brass and other copper alloys. However, it should be added in amounts just sufficient to remove impurities and should not be present in the alloy.

Aluminum.—The addition of small amounts of aluminum to brass appears to be beneficial in deoxidizing, in retarding oxidation, and in reducing the volatilization of zinc at elevated temperatures. The aluminum content should be kept below 0.20 per cent.

Nickel.—The effect of nickel on brass is to improve its mechanical properties and corrosion resistance. The well-known nickel-silver and similar high nickel-copper-zinc alloys containing up to 18 per cent nickel are being die cast with excellent results.

Silicon.—Silicon in copper-zinc alloys for brass die casting has been found to be highly beneficial. It lowers the melting point, increases fluidity, and tends to counteract and diminish the deposit of zinc oxide on the die surfaces. The die surfaces remain clean and require little or no die lubricant, to allow free ejection of the casting from the die. Silicon also greatly improves the physical properties as is shown below.

The effect of other elements, chromium, tungsten, molybdenum, vanadium, cobalt, and titanium, on brass alloys for die casting are now being studied.

As the copper content of the copper-zinc series of alloys increases there is a steady rise in melting point temperature as shown in Table I. From this table it can be seen that the die casting of brasses with copper content greater than 70 per cent becomes increasingly difficult. For example, the 80 per cent copper, 20 per cent zinc alloy, has a melting point of 1846 F. This alloy is much more severe on dies than is the 60–40 composition with a melting point of 1655 F. By replacing approximately 4 per cent of the zinc content of the 80 per cent copper, 20 per cent zinc alloy with the same amount of silicon, the Tombasil[1] type alloy is obtained. This alloy has a melting point somewhat lower than that of the 60–40 alloy. It has a minimum tensile strength of 85,000 lb. per sq. in., an elongation of about 10 per cent, and has an average Brinell hardness of 170. The corrosion resistance of this alloy is very high, and experiments have shown that it is a valuable bearing metal. Its high fluidity makes it an excellent casting alloy.

TABLE II.—NOMINAL COMPOSITION AND PROPERTIES OF BRASS DIE CASTING ALLOYS

Copper, per cent	57.0 to 59.0	63.0 to 65.0	80.0 to 81.5	42.0[a]
Zinc, per cent	40.0 to 42.0	33.0 to 35.0	13.5 to 16.0	41.0
Tin, per cent	0.50 to 1.50	....	....	....
Silicon, per cent	....	0.75 to 1.25	3.75 to 4.25	....
Lead, per cent	....	....	....	1.0
Nickel, per cent	....	....	....	16.0
Tensile strength, lb. per sq. in.	55 000 to 60 000	65 000 to 70 000	85 000 to 95 000	85 000 to 95 000
Yield point, lb. per sq. in.	30 000 to 35 000	35 000 to 40 000	65 000 to 70 000	65 000 to 72 000
Elongation, in 2 in., per cent	15 to 20	20 to 30	8 to 12	10 to 20
Reduction of area, per cent	15 to 20	15 to 20	10 to 15	10 to 18
Brinell hardness	120 to 130	120 to 130	160 to 180	160
Specific gravity	8.47	8.50	8.15	8.45
Color	yellow	yellow	light yellow	white
Machinability	fair	fair	hard	fair
Corrosion resistance	good	good	high	high
Bearing properties	poor	fair	good	poor

[a] According to W. W. Sieg, Titan Metal Co.

Another good die casting alloy is one composed of 65 per cent copper, 34 per cent zinc and 1 per cent silicon. This alloy in the pressure die cast state will show a tensile strength around 70,000 lb. per sq. in. and an elongation of about 30 per cent.

The white nickel brass die castings also are capable of high physical properties, in addition to possessing good corrosion resistance.

Table II shows the nominal composition and physical properties of some of the standard brass die casting alloys.

ALLOYING PRACTICE

Particular care should be exercised in the preparation of brass die casting alloys. The rate of melting, the purity of the metals, and types of furnaces used all have an influence on the physical properties of the alloy. These factors must be carefully regulated to minimize the tendency for gas absorption, formation of oxides, and inequalities of properties, usually accompanying the lack of control. Liberal use should be made of suitable fluxes to exclude oxygen and other gases and to combine with oxides and impurities in the melt. When the alloy is cast in the plastic or semi-liquid state, close to the melting point, there is a great possibility for oxides and foreign inclusions to be intermixed with the melt, which ultimately are to be found in the castings. Obviously such non-metallic substances adversely affect machinability in that fine cutting edges of tools may be readily dulled. By using the alloy in the completely liquid state, there is a much greater chance of keeping these oxides and impurities out of the melt, since they will rise to the surface and can be removed by skimming.

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[1] Three alloys of this type are available: Tombasil Alloy, Ajax Metal Co.; Brastil Alloy, Doehler Die Casting Co.; and Webert Alloy, American Brass Co.

DIE LUBRICANT

An essential item in connection with brass die casting is the material used for coating and lubricating die parts. A good die lubricant is necessary to act as a protective film between the hot metal and the die surfaces, and to act as a lubricant for cores and other movable parts. A good film materially reduces the rate of heat transmission to the die surfaces. A water suspension of colloidal graphite has been found to give good results, and is being used consistently for this purpose.

THE ECONOMIC POSITION OF BRASS DIE CASTINGS

The brass die casting process has proven sufficiently economical and practical to be recognized as a commercial means of manufacture. Brass die castings can assume a rightful place along with die castings of other alloys as well as other methods for the fabrication of metals.

The number and variety of applications of brass die castings are steadily increasing. It is not likely, however, that they will replace altogether products of other methods. It is believed that there is a specific field for each process, and for each type of alloy.

Brass die castings have not as yet been able to compete with simple shaped brass sand castings, which require little or no machining. However, there are many parts formerly sand cast which are now being economically die cast. This is due to the savings brought about by reduction in weight and in the lower finishing costs of brass die castings. For the same alloy brass die castings show superior mechanical properties to sand casting as a result of finer grain size. The surface finish and soundness throughout in brass die castings are superior to sand castings. Sharper detail and greater dimensional accuracy can be had with die castings, and thinner wall sections can be die cast than are possible with sand castings.

With the development of the brass die casting process competition between brass die castings and brass forging is growing keener. Die castings have greater accuracy and finer detail, while forgings afford greater ease of machinability and greater soundness of structure. For a given alloy, the basic metal cost for forging is much higher than that for the die cast alloy, due chiefly to the number of operations in preparing the metal for the final forging operation.

Brass castings have the inherent porosity of die castings, and low as this porosity may be, the castings are not comparable in soundness or density with forgings. While die castings will not replace forgings entirely there is hardly a doubt that many parts now forged will be die cast at a substantial saving.

Brass die castings will replace zinc die castings only where the application requires the high strength, the corrosion resistance, bearing properties and permanence of brass. For uses where zinc die castings have proven satisfactory over long periods of service, it is doubtful whether brass die castings can ever expect to overcome the many advantages of zinc and to replace it. The lower cost of the zinc alloy, the lower die cost and greater die life, and the high-speed production of present zinc die castings make it almost impossible for brass die castings to compete.

The die casting of brass, nickel-silver, and similar copper alloys is making substantial progress. Further developments are expected to bring the process into relationship to other methods for fabricating metals.

APPENDIX II

A STUDY OF DIE DESIGN CHANGES FOR THE IMPROVEMENT OF THE SOUNDNESS AND UNIFORMITY OF TEST BARS

By G. L. Werley[1]

This paper is divided into two parts: Part I pertaining to the development of an insert or unit die to produce sound test bars of uniform strength, and Part II covering similar tests on a die which was built in 1927 primarily to cast test specimens for the first exposure tests conducted by Committee B-6 on Die Cast Metals and Alloys. This die is referred to herein as the A.S.T.M. die.

The work under Part I was done in the interests of reducing the number of specimens required to be averaged to obtain significant results for routine tests. Part II was undertaken to effect similar improvements in the A.S.T.M. die so that those producers who have this type of die and are cooperating in the production of test bars for the proposed A.S.T.M. atmospheric exposure test can readily alter their die to attain an improvement in uniformity and soundness of specimens.

PART I

In 1932, a unit or insert die provided with inserts for casting various types of test specimens was put into operation.

Specimens cast in the unit for the production of ¼-in. test bars, under carefully controlled conditions, exhibited a wide degree of nonuniformity in physical properties. A statistical analysis showed that forty such impact bars were required to obtain, in 95 out of 100 cases, an average within ±10 per cent of the average of a very large number of specimens. Radiographs also showed the test bars to have considerable porosity.

In an attempt to produce sound test bars of uniform strength, small changes were made repeatedly in the gates, vents, and overflow wells. Collectively, these changes produced the desired results and in addition each die change, which increased the uniformity, also increased the impact strength and, to a lesser extent, the Brinell hardness. The tension bars were improved in soundness but not in strength. In fact, a slight loss in tensile strength resulted.

Changes were made in the die with the following objectives in view:

1. The castings were to be virtually free of porosity in the test sections as shown by radiographs.
2. The impact bars were to have the same strength for the gate and vent halves of the specimen when tested at 21 C.
3. The average impact strength of any five bars taken at random was to be within ±10 per cent of the average of a large number of specimens in 95 out of 100 cases.

The original design of the unit die for the production of ¼-in. test bars is shown in Fig. 1. When this die was put into service the ¼-in. impact specimens cast were too porous to be considered satisfactory. In order to cast sounder specimens, the following changes were made in the die:

1. The overflow well at the vent end of the impact cavity was increased in size.

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[1] New Jersey Zinc Co., Palmerton, Pa.

2. Four vents were added to the die at regularly spaced distances along the side of the impact cavity (see Fig. 8).

3. A waster in the shape of a $\frac{5}{16}$-in. square impact bar, with an accompanying overflow well, was added to the die between the existing round tension and $\frac{1}{2}$-in. square impact cavities to facilitate getting the die up to the proper temperature and to assist in holding the temperature at a constant value (see Fig. 8).

The above changes were made in the die prior to the intensive study of the optimum design of this die.

TABLE I.—PHYSICAL PROPERTIES OF ZAMAK 3 CAST IN THE UNIT DIE.

Date cast: December 12, 1935.
Pressure: 1500 lb. per sq. in. (calculated).
Metal temperature: 425 C.
Die temperature: 150, 200, and 250 C.

	150 C.	200 C.	250 C.
Tensile strength, lb. per sq. in.	44 000	43 400	43 000
Impact strength (top or gate end), ft-lb.[a]	35	35	38
Standard deviation, ft-lb.[b]	2.4	3.5	4.0
Coefficient of variation[c]	6.9	9.9	10.5
Impact strength (bottom or vent end), ft-lb.[a]	7.3[d]	9.6	18
Standard deviation, ft-lb.	2.2	3.1	5.3
Coefficient of variation	30.4	31.8	29.2
Impact strength, grand average, ft-lb.	21	22	28
Standard deviation, ft-lb.	13.8	13.2	10.7
Coefficient of variation	65.8	59.6	38.4

[a] Tested at 21 ± 0.5 C.

[b] Calculated using formula $\sqrt{\frac{\Sigma x^2}{n}}$, where x is the deviation of the individual bars from the arithmetic average and n is the number of specimens.

[c] The coefficient of variation is a measure of the variability which is independent of the scale units and which takes into account the size of deviations relative to the mean.

The coefficient of variation is determined by using the formula $V = \frac{100\,\sigma}{M}$ where σ = the standard deviation and M = the arithmetic mean.

[d] The impact values were determined on a Charpy type machine fitted with three interchangeable hammers having the following capacities: 0 to 10.3; 0 to 38.5; 0 to 103.6 ft-lb. Contrary to our usual practice, impact values under 15 ft-lb. were reported to one decimal place. However, such results are believed to be significant when the proper hammer is used.

NOTE.—Through common usage, the gate and vent ends are sometimes referred to as "top" and "bottom," respectively. Therefore, throughout this report wherever "top" is used, it refers to the gate end; and "bottom" refers to the vent end.

TABLE II.—PHYSICAL PROPERTIES OF ZAMAK 3 CAST IN THE IMPROVED UNIT DIE.

Date cast: March 4, 1936.
Pressure: 1500 lb. per sq. in. (calculated).
Metal temperature: 415 C.
Die temperatures: 150, 160, 170, 180, 190, 200, 210, 220, 230, 240, and 250 C.

	150 C.	160 C.	170 C.	180 C.	190 C.	200 C.	210 C.	220 C.	230 C.	240 C.	250 C.
Tensile strength, lb. per sq. in.	44 000	42 600	41 900	40 500	40 700	40 800	41 300	41 100	40 500	40 500	40 100
Impact strength (top or gate end), ft-lb.[a]	38	39	40	40	42	42	42	44	42	41	38
Standard deviation, ft-lb.	3.1	3.0	5.0	4.6	1.8	2.4	1.2	0.6	3.6	4.1	3.3
Coefficient of variation	8.1	7.6	12.5	11.5	4.3	5.8	2.9	1.3	8.7	10.1	8.7
Impact strength (bottom or vent end), ft-lb.[a]	36	38	45	42	44	44	43	44	43	43	43
Standard deviation, ft-lb.	4.2	4.5	0.8	3.1	0.7	0.7	0.7	0.6	0.6	0.8	0.5
Coefficient of variation	11.7	12.0	1.9	7.3	1.6	1.7	1.7	1.3	1.3	2.0	1.2
Impact strength, grand average, ft-lb.	37	38	43	41	43	43	43	44	42	42	40
Standard deviation, ft-lb.	3.8	3.9	4.3	4.1	1.8	2.0	1.1	0.6	3.1	3.2	3.3
Coefficient of variation	10.4	10.2	10.0	10.0	4.2	4.6	2.6	1.3	7.2	7.6	8.1

[a] Tested at 21 ± 0.5 C.

The die with the above construction changes produced Zamak 3 bars (A.S.T.M. alloy No. XXIII)[1] with the physical properties given in Table I when cast in a modern die casting machine.

The data in Table I show that, in impact strength, the vent halves of the bars are greatly inferior to the gate halves as well as being less uniform in strength.

Radiographs, Figs. 2, 3, and 4, show considerable porosity at the gate, and some porosity at the vent, of the impact bars. Radiographs, Figs. 5, 6, and 7, show considerable porosity in the vent end, and some objectionable porosity in the test section, of the round tension bars.

In Fig. 8 is shown the net result of the changes made in the die from its original construction, as shown in Fig. 1. It now embodies what is believed to be about the optimum design for the die casting of Zamak alloys in this particular insert die.

As previously stated, the die changes were made in individual small steps. In this manner it was possible to study the effects which each die change produced and, therefore, recognize when the optimum condition for each type of change was reached. It is known that if the gate opening, for instance, is at its optimum size, a change in the size of the overflow well may disturb the optimum condition established for the gate. Thus, any change made in the die may cause some former alteration to be out of balance and create an almost endless series of minor changes. The die as shown in Fig. 8 met the requirements listed earlier concerning uniformity and strength and, therefore, was considered as being correctly designed.

The changes made in the die can be seen by comparing Figs. 1 and 8. The major changes consisted of the following:

1/4-in. Impact Bar Cavity:

1. Gate depth increased from 0.030 in. to 0.083 in.
2. Overflow well runner depth increased from 0.030 in. to 0.088 in.
3. Overflow well increased from 0.07 cu. in. to 0.6 cu. in.
4. Four vents were placed in the die along the length of the cavity.

1/4-in. Round Tension Bar Cavity:

1. Gate depth increased from 0.030 in. to 0.075 in.
2. Overflow well increased from 0.06 cu. in. to 0.15 cu. in.[a]

Further Alterations:

1. The addition of a $\frac{5}{16}$-in. square impact bar cavity[b] having:
    (*a*) Gate depth of 0.090 in.
    (*b*) Overflow well runner depth of 0.080 in.
    (*c*) 0.5-cu. in. overflow well.
    (*d*) Vent from the overflow well of 0.007-in. depth.

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[a] The vent end grip of a tension bar is in effect an overflow well so far as the test section is concerned. On this basis, the overflow well capacity is 0.3 cu. in. plus the above-mentioned 0.15 cu. in. or a total of 0.45 cu. in.

[b] This casting has no particular value as a test specimen and was added only to facilitate getting the die up to the proper temperature and to assist in holding this temperature at a constant value.

Zamak 3 (A.S.T.M. alloy No. XXIII)[2] had the physical properties given in Table II when cast in the die shown in Fig. 8 and using a modern die casting machine.

Figure 9 shows the impact data of Tables I and II. By means of this chart the improvement in impact strength resulting from the die changes are readily apparent.

It will be noticed that the strength of the top half of the impact bars was not greatly affected by the die changes. The greatest improvement was in the impact strength of the bottom half of the bar. In many cases, in the final cast, the vent end of the bar exceeded in impact strength the gate end although the difference between the two was not considered significant.

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[1] Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 – 34 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 706 (1934); also 1936 Book of A.S.T.M. Tentative Standards, p. 356.

[2] *Loc. cit.*

The die was now in such shape that one of the objectives of this experiment had been attained, namely, that the impact bars should possess the same strength for the gate and vent halves of the specimen at 21 C.

The radiographs, Figs. 10 and 11, show the impact bars to be virtually free of porosity. Figures 12, 13, and 14 are radiographs of round tension bars. While these bars show the presence of porous areas in the grip ends, the test sections are in most cases free of porosity. It is believed that the porosity of these bars could have been reduced further if there had been room in the insert die to increase the overflow well. The second objective was accordingly met, that the bars should be virtually free of porosity.

In order to study the uniformity of the specimens, 100 impact bars of Zamak 3 cast under controlled conditions were tested for their impact strength at both the gate and vent ends, making a total of two hundred tests, with the following results:

IMPACT STRENGTH, FT-LB.	NUMBER OF PIECES
33 to 35	2
37 to 39	4
39 to 41	7
41 to 43	11
43 to 45	20
45 to 47	153
47 to 49	2
49 to 51	1
Total	200

A statistical analysis of these data showed that a sample size of 3 to 5 bars was sufficient to yield an average impact value within ±10 per cent of the true average in 95 out of 100 cases, which demonstrated that the third condition for test bars had been fulfilled.

It is known that Zamak 3 as normally cast does not show any appreciable drop in impact strength during a 95 C. steam test of 10 days' duration. In order to determine whether this characteristic resistance to steam was retained in the specimens cast in the altered insert die, test bars were cast at a number of die temperatures and exposed in the 95 C. steam tank, with the results given in Table III.

From these data, it will be seen that while the impact strengths for as-cast specimens are satisfactory for all of the die temperatures tried, namely, 150 to 250 C. in 10 deg. Cent. intervals, nevertheless, the steam test limits the permissible die temperature to a range of 150 to 220 C.[1] Above 220 C. die temperature, there is an appreciable drop in impact strength during the 10-day 95 C. steam test and the impact values show wide variations as evidenced by the large coefficient of variations.

The die was considered to be in its optimum condition, so far as could be determined at this time, for Zamak 3, and, further tests along similar lines on three other Zamak alloys (including A.S.T.M. alloys Nos. XXI and XXV) showed results as favorable as those obtained for Zamak 3. Only the data for Zamak 5 are reported here.

The results for Zamak 5 (A.S.T.M. alloy No. XXV) are included in this paper to link Part I with Part II since Zamak 5 was used in the development of the A.S.T.M. die (Part II of this report).

It would have been desirable to conduct the development of the insert die and the A.S.T.M. die with the same alloy. However, this was not practical, if the alloy composition changes in the die casting pot were to be held to a minimum to avoid contamination of one alloy with another, since the development work extended from December 12, 1935, to November 4, 1936, and was conducted in connection with many other experimental programs. It so happened that Zamak 3 was the alloy on which experimental work was in progress when the insert die was altered and Zamak 5 was being studied when the A.S.T.M. die was being improved.

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[1] The permissible die temperature range may be different for each die since there is no standardized procedure for the determination of die temperatures.

TABLE III.—THE EFFECT OF STEAM TREATMENT ON THE PHYSICAL PROPERTIES OF ZAMAK 3 CAST IN THE IMPROVED UNIT DIE.

Date cast: March 4, 1936.
Pressure: 1500 lb. per sq. in. (calculated).
Metal temperature, 415 C.
Die temperatures: 150, 160, 170, 180, 190, 200, 210, 220, 230, 240, and 250 C.

	150 C.	160 C.	170 C.	180 C.	190 C.	200 C.	210 C.	220 C.	230 C.	240 C.	250 C.
AS CAST											
Impact strength (top or gate end), ft-lb., tested at 21 ± 0.5 C.	38	39	40	40	42	42	42	44	42	41	38
Coefficient of variation	8.1	7.6	12.5	4.5	4.3	2.0	2.9	1.3	8.7	10.1	8.7
Impact strength (bottom or vent end), ft-lb., tested at 21 ± 0.5 C.	36	38	45	42	44	44	43	44	43	43	43
Coefficient of variation	11.7	12.0	1.9	7.3	1.6	1.7	1.7	1.3	1.3	2.0	1.2
EXPOSED IN 95 C. STEAM TEST FOR 10 DAYS											
Impact strength (top or gate end), ft-lb., tested at 21 ± 0.5 C.	43	43	43	42	40	40	42	37	35	21	25
Coefficient of variation	6.2	7.7	2.3	5.7	10.0	9.2	3.8	17.7	29.1	57.5	59.2
Impact strength (bottom or vent end), ft-lb., tested at 21 ± 0.5 C.	44	44	42	43	43	43	42	42	32	29	30
Coefficient of variation	6.0	1.6	8.0	2.1	0.9	2.8	1.2	3.3	30.4	51.4	38.3

TABLE IV.—IMPACT STRENGTH DATA OF ZAMAK 5 CAST IN THE IMPROVED UNIT DIE.

Date cast: March 12, 1936.
Pressure: 1500 lb. per sq. in. (calculated).
Metal temperature: 410 C.
Die temperatures: 150, 160, 170, 180, 190, 200, 210, 220, 230, 240, and 250 C.

SPECIMENS CAST AFTER FINAL ALTERATIONS TO INSERT DIE—AS CAST

	150 C.	160 C.	170 C.	180 C.	190 C.	200 C.	210 C.	220 C.	230 C.	240 C.	250 C.
Impact strength (top or gate end), ft-lb.[a]	42	44	44	48	49	49	47	48	47	48	46
Coefficient of variation	4.1	5.5	5.3	4.4	1.4	0.8	4.0	0.8	4.1	1.9	1.5
Impact strength (bottom or vent end), ft-lb.[a]	33	43	44	45	48	48	48	48	47	48	46
Coefficient of variation	13.6	9.0	12.0	11.2	1.3	2.7	0.8	0.8	1.7	2.7	1.9

[a] Tested at 21 ± 0.5 C.

The data on Zamak 5 show that the insert die was equally capable of casting this alloy to meet the standards established for Zamak 3, namely, (1) that the castings be virtually free of porosity in the test sections as revealed by radiographs, (2) that the impact bars show approximately the same strength for the gate and vent halves of the specimen when tested at 21 C.

The impact data obtained on Zamak 5 cast in the altered insert die are given in Table IV and radiographs of these specimens are shown in Figs. 15, 16, 17, 18, and 19.

PART II

The work done to improve the A.S.T.M. die was started following the meeting of Committee B-6 held at Atlantic City on July 1, 1936. At that meeting, the possibility of producing sound test bars of uniform and higher strength than previously secured in an insert die was reported. On the basis of this report it was agreed that for the ensuing exposure tests the dies to be used by the producers should, if possible, be altered to produce sounder and more uniform specimens than were used in the first series of tests. Since some of the specimens will probably be produced on A.S.T.M. and not insert type dies, it became necessary to try to improve the A.S.T.M. die to the same standard attained in the unit die.

The A.S.T.M. die in its original form is shown in Fig. 20. The flat tension bar cavity was altered during the development work but the results are not discussed here because Committee B-6 has standardized exclusively on the use of the round bar for tension and elongation tests. It is recommended, however, that the flat tension bar cavity be retained since the casting of some such form of waster bar in this part of the die is believed to be essential.

TABLE V.—PHYSICAL PROPERTIES OF ZAMAK 5 CAST IN THE A.S.T.M. DIE.

Date cast: July 16, 1936.
Pressure: 1500 lb. per sq. in. (calculated).
Metal temperature: 410 C.
Die temperatures: 150, 160, 170, 180, 190, 200, 210, 220, 230, 240, and 250 C.

	150 C.	160 C.	170 C.	180 C.	190 C.	200 C.	210 C.	220 C.	230 C.	240 C.	250 C.
Tensile strength, lb. per sq. in.	48 800	48 300	48 800	47 900	48 000	47 300	47 200	47 500	46 500	47 200	45 000
Impact strength (top or gate end), ft-lb.[a]	19	20	22	21	22	21	27	29	28	30	27
Standard deviation, ft-lb.	2.9	2.1	1.5	2.9	2.4	2.2	6.0	6.0	3.7	5.1	5.5
Coefficient of variation	15.5	10.5	6.9	14.0	11.0	10.7	21.8	20.4	13.4	17.2	20.0
Impact strength (bottom or vent end), ft-lb.[a]	17	17	19	21	20	28	28	26	28	32	32
Standard deviation, ft-lb.	5.2	2.3	2.4	7.1	2.1	5.7	5.5	4.0	5.9	6.4	7.2
Coefficient of variation	31.6	14.1	13.0	33.7	10.8	20.3	19.9	15.4	20.9	20.1	22.6
Impact strength, grand average, ft-lb.	18	18	20	21	21	24	28	28	28	31	30
Standard deviation, ft-lb.	4.4	2.7	2.5	5.4	2.6	5.6	5.8	4.9	4.9	5.9	6.7
Coefficient of variation	24.9	15.0	12.3	26.0	12.4	23.0	20.9	17.5	17.4	19.1	22.7

[a] Tested at 21 ± 0.5 C.

Specimens cast in this die, under carefully controlled conditions, exhibited a wide degree of nonuniformity in physical properties, and radiographs showed that the impact bars had considerable porosity.

The A.S.T.M. die in its original form shown in Fig. 20 when operated in a modern die casting machine produced Zamak 5 test bars with the physical properties given in Table V.

The data in Table V show that the two halves of the impact bars were about equal in strength but the specimens were not uniform nor did they possess the high impact values obtainable in the improved or altered insert die.

Radiographs, Figs. 21 and 22, show very objectionable porosity at the vent end and fine voids throughout the remainder of the bars. Radiographs, Figs. 23, 24, and 25, show the round tension bars to be free of porosity in the test section and also throughout the remainder of the bars except for some minor voids in the vent end.

The same line of attack used to produce sound, uniform strength test bars in the insert die was followed in the A.S.T.M. die, namely, making repeated changes in the gates, vents and overflow wells. As in the case of the insert die, the impact strength, and to a lesser extent, the Brinell hardness increased with the uniformity of the bars. Again, there was a slight loss in tensile strength as a result of these die changes.

In Fig. 26 are shown the net result of the changes made in the die from its original construction shown in Fig. 20. These changes produced material improvements in the impact strength, uniformity, and soundness of the bars but failed to produce the high degree of perfection attained in the insert die.

Lack of time has prevented completion of this investigation. The changes in the gate and vent openings, the addition to or enlarging of overflow wells, and the inclusion of waster bars were established, by trial, to be at about their optimum. It is believed that to achieve the same degree of perfection in this die as had been accomplished in the insert die it would be necessary to alter the major construction of this die. Such an operation would be time-consuming and since several die casters have already requested the information on the die changes so that they can alter dies or build new ones for the production of test bars for the proposed A.S.T.M. atmospheric exposure tests, it was decided to forego any further alteration to the die at this time.

The changes made in the die can be seen by comparing Figs. 20 and 26. The major changes consisted of the following:

1/4-in. Impact Bar Cavity:

1. Gate depth increased from 0.038 in. to 0.070 in. During the tests, gates up to 0.100 in. were tried.
2. Overflow well added and increased until it had a capacity of 0.9 cu. in.
3. Runner depth to overflow well increased from 0.030 in. to 0.070 in.

1/4-in. Round Tension Bar Cavity:

1. Gate depth increased from 0.046 in. to 0.075 in.
2. Overflow well of 0.07 cu. in. capacity added.
3. Runner depth to overflow well increased from 0.030 in. to 0.048 in.

Further Alterations:

1. A 3 in. long by $\frac{3}{4}$ in. wide by $\frac{1}{4}$ in. deep cavity was cut along the outer edge of the die next to the impact cavity.[a]
2. The gate runners were increased in thickness by $\frac{1}{8}$ in. from the sprue to the gates of the tension, impact, and waster bar cavities.

---

[a] This casting has no particular value as a test specimen. It was added to the die for the purpose of increasing the strength uniformity of the impact bar by facilitating the maintenance of a more uniform die temperature.

The die when altered as shown in Fig. 26 produced Zamak 5 test bars with the physical properties given in Table VI.

Figure 27 shows the impact data of Tables V and VI. By means of this chart the improvement in impact strength resulting from the die changes made during the period of July 16, 1936, to November 4, 1936, are readily apparent.

It will be noticed that the impact strength was almost doubled as a result of the die changes. Unfortunately, however, the gate and vent ends of the impact bar differed materially in impact strength and all of the die changes which were tried increased, rather than decreased, the spread in strength between the two ends of the bars.

The radiographs, Figs. 28 and 29, show the impact bars to be virtually free from porosity and Figs. 30[1] and 31[1] show only minor voids in the vent end of the round tension bars. Again, it is believed that the porosity in the round tension bars could have been reduced had there been room in the die further to increase the overflow well.

---

[1] As in all former cases, five round tension bars were cast at 10 deg. Cent. intervals of die temperature from 150 to 250 C. but only three specimens of each group, and none of the 200 C. die temperature cast, were radiographed. This economy was practiced in order to get the tension bars on two plates.

It will be seen in Table VII that the impact strengths for as-cast specimens are satisfactory for all die temperatures tried, namely, 150 to 250 C. in 10 deg. Cent. intervals.

For steam exposed specimens the coefficients of variation are all high values with no indication that any restricted range of die temperature is any better than the entire range of 150 to 250 C.

TABLE VI.—PHYSICAL PROPERTIES OF ZAMAK 5 CAST IN THE IMPROVED A.S.T.M. DIE.

Date cast: November 4, 1936.
Pressure: 1500 lb. per sq. in. (calculated).
Metal temperature: 410 C.
Die temperatures: 150, 160, 170, 180, 190, 200, 210, 220, 230, 240, and 250 C.

	150 C.	160 C.	170 C.	180 C.	190 C.	200 C.	210 C.	220 C.	230 C.	240 C.	250 C.
Tensile strength, lb. per sq. in.	49 100	48 500	49 200	46 700	46 700	45 700	45 600	44 700	44 400	44 500	43 800
Impact strength (top or gate end), ft-lb.[a]	33	33	35	34	34	36	35	40	39	38	38
Standard deviation, ft-lb.	2.6	2.8	3.0	2.4	1.7	2.6	2.2	2.0	3.2	3.1	1.7
Coefficient of variation	7.9	8.4	8.7	7.1	5.1	7.2	6.3	5.1	8.2	8.0	4.5
Impact strength (bottom or vent end), ft-lb.[a]	47	48	47	46	50	50	51	49	49	48	49
Standard deviation, ft-lb.	3.2	2.7	1.2	1.4	0.7	0.6	0.7	0.9	0.7	0.7	0.9
Coefficient of variation	6.8	5.7	2.5	2.9	1.3	1.3	1.4	1.8	1.5	1.5	1.8
Impact strength, grand average, ft-lb	40	40	41	40	42	43	43	44	44	43	43
Standard deviation, ft-lb.	7.7	7.9	6.4	6.5	8.1	6.9	8.0	5.0	6.0	5.3	5.3
Coefficient of variation	19.0	19.5	15.7	16.1	19.3	16.0	18.6	11.2	13.7	12.3	12.2

[a] Tested at 21 ± 0.5 C.

TABLE VII.—THE EFFECT OF STEAM TREATMENT ON THE PHYSICAL PROPERTIES OF ZAMAK 5 CAST IN THE IMPROVED A.S.T.M. DIE.

Date cast: November 4, 1936.
Pressure: 1500 lb. per sq. in. (calculated).
Metal temperature: 410 C.
Die temperatures: 150, 160, 170, 180, 190, 200, 210, 220, 230, 240, and 250 C.

	150 C.	160 C.	170 C.	180 C.	190 C.	200 C.	210 C.	220 C.	230 C.	240 C.	250 C.
SPECIMENS CAST AFTER FINAL ALTERATIONS TO DIE—AS CAST											
Impact strength (top or gate end), ft-lb.[a]	33	33	35	34	34	36	35	40	39	38	38
Coefficient of variation	7.9	8.4	8.7	7.1	5.1	7.2	6.3	5.1	8.2	8.0	4.5
Impact strength (bottom or vent end), ft-lb.[a]	47	48	47	46	50	50	51	49	49	48	49
Coefficient of variation	6.8	5.7	2.5	2.9	1.3	1.3	1.4	1.8	1.5	1.5	1.8
SIMILAR SPECIMENS EXPOSED IN 95 C. STEAM TEST FOR 10 DAYS											
Impact strength (top or gate end), ft-lb.[a]	6.9	5.1	4.2	6.2	6.0	8.8	8.7	5.3	7.9	5.2	5.3
Coefficient of variation	59.0	58.2	44.3	82.4	64.0	75.9	58.6	58.7	64.8	79.2	56.2
Impact strength (bottom or vent end), ft-lb.[a]	13.7	17.0	10.1	11.9	5.0	10.3	14.8	6.5	8.6	11.3	8.2
Coefficient of variation	25.0	46.2	41.1	59.1	41.8	70.1	43.1	90.5	89.7	63.1	73.3

[a] Tested at 21 ± 0.5 C.

It is assumed from these data that the die temperature casting range for Zamak 5 is apparently not greatly restricted.

CONCLUSION

It is clearly evident that the insert die has been altered to a higher degree of perfection than the A.S.T.M. die. It would, therefore, appear desirable that any new dies constructed for use in producing specimens for the proposed A.S.T.M. atmospheric exposure tests be insert dies.

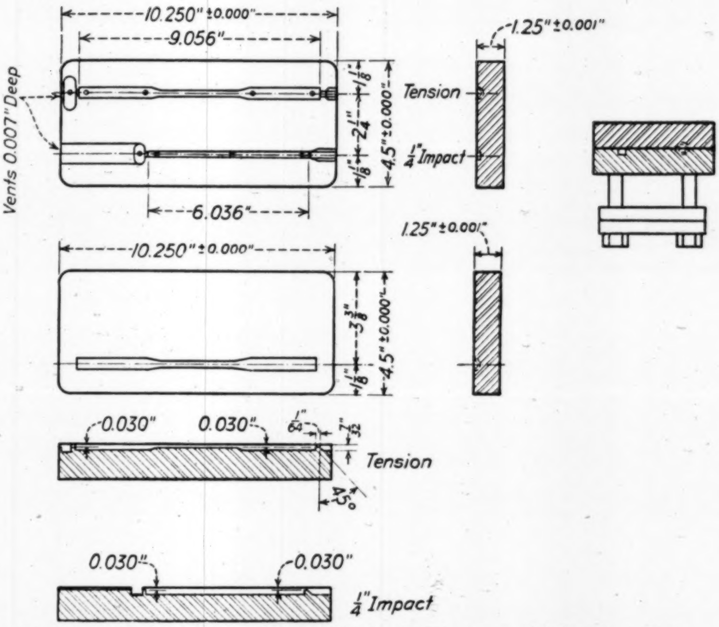
10.250" ±0.000"
9.056"
1.25" ±0.001"
Vents 0.007" Deep
Tension
¼" Impact
1⅛"
2¼"
1⅛"
4.5" ±0.000"
6.036"
1.25" ±0.001"
10.250" ±0.000"
3⅜"
1⅛"
4.5" ±0.000"
0.030"
0.030"
1/64"
7/32"
Tension
45°
0.030"
0.030"
¼" Impact

FIG. 1.—Original Plan of Unit Die for Production of ¼-in. Test Bars.

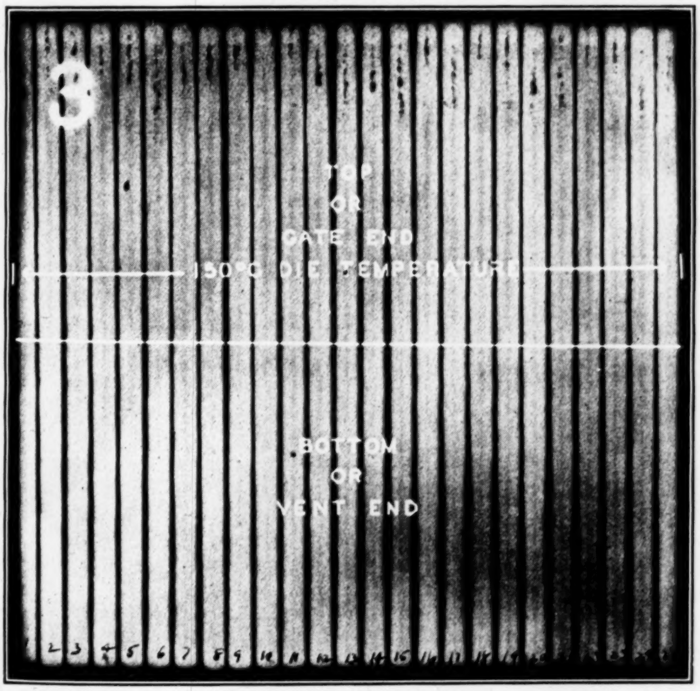
3
TOP
OR
GATE END
150°C DIE TEMPERATURE
BOTTOM
OR
VENT END
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19

FIG. 2.—Radiograph of ¼-in. Impact Bars of Zamak 3 Cast at 150 C. in the Unit Die.

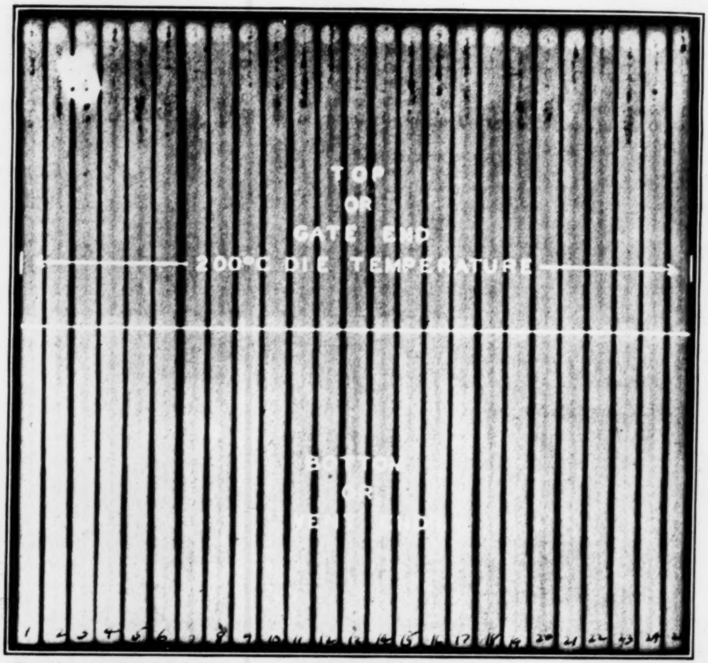
TOP
OR
GATE END
200°C DIE TEMPERATURE
BOTTOM
OR
VENT END
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25

FIG. 3.—Radiograph of $\frac{1}{4}$-in. Impact Bars of Zamak 3 Cast at 200 C. in the Unit Die.

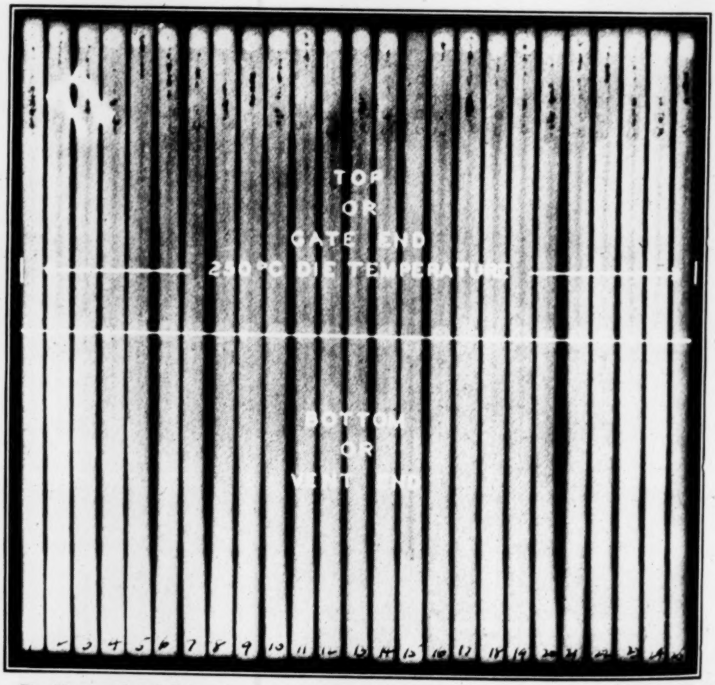
TOP
OR
GATE END
250°C DIE TEMPERATURE
BOTTOM
OR
VENT END
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25

FIG. 4.—Radiograph of $\frac{1}{4}$-in. Impact Bars of Zamak 3 Cast at 250 C. in the Unit Die.

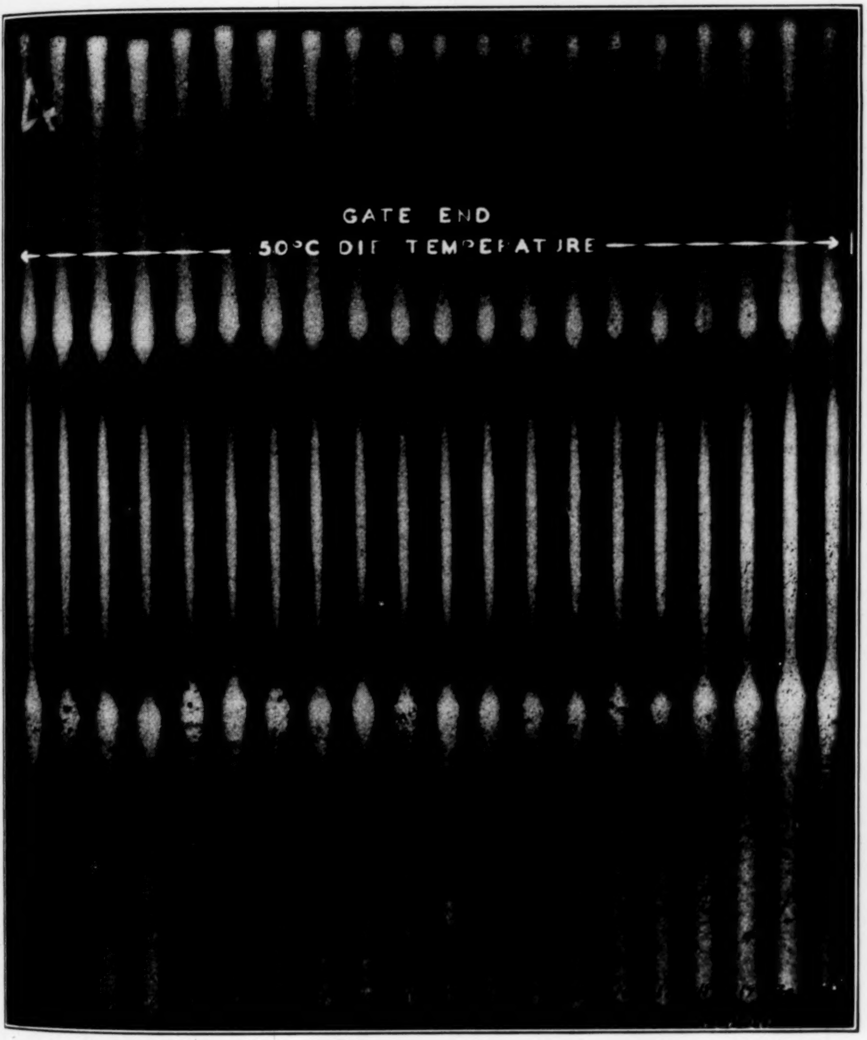
GATE END
50°C DIE TEMPERATURE

FIG. 5.—Radiograph of $\frac{1}{4}$-in. Round Tension Bars of Zamak 3 Cast at 150 C. in the Unit Die.

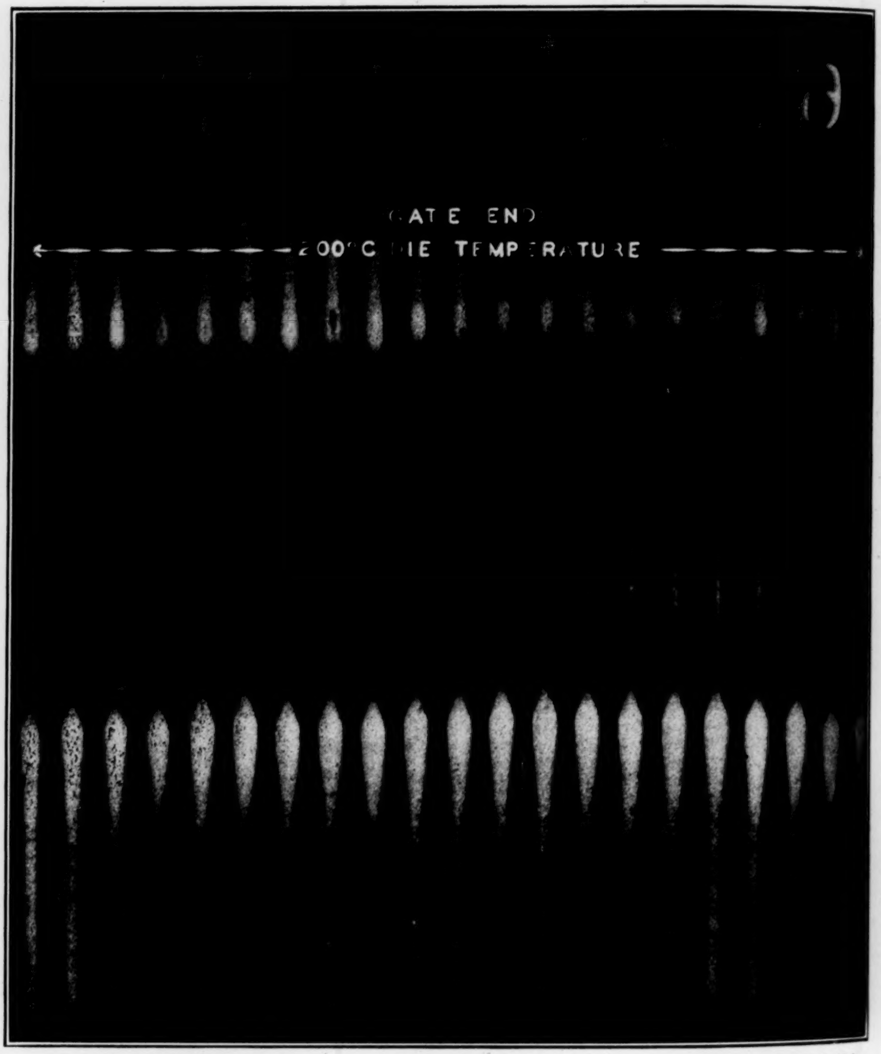
GATE END
200°C DIE TEMPERATURE

FIG. 6.—Radiograph of ¼-in. Round Tension Bars of Zamak 3 Cast at 200 C. in the Unit Die.

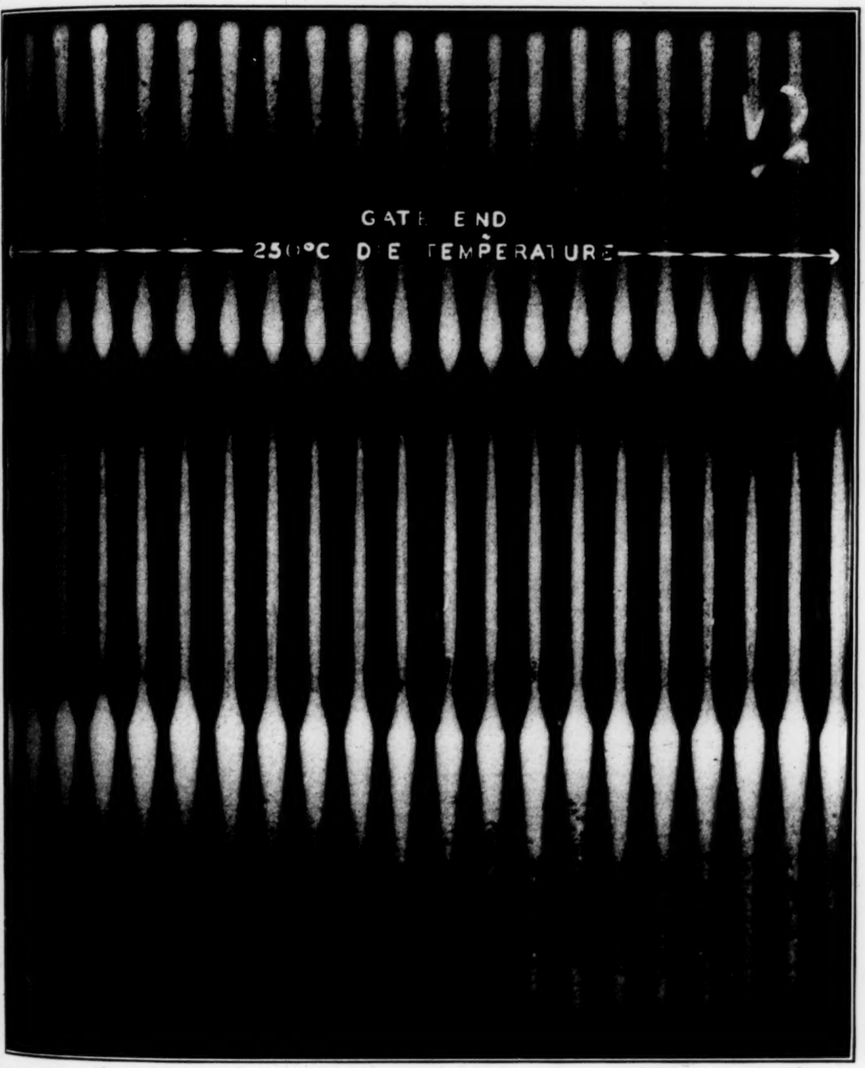
GATE END
250°C DIE TEMPERATURE

FIG. 7.—Radiograph of ¼-in. Round Tension Bars of Zamak 3 Cast at 250 C. in the Unit Die.

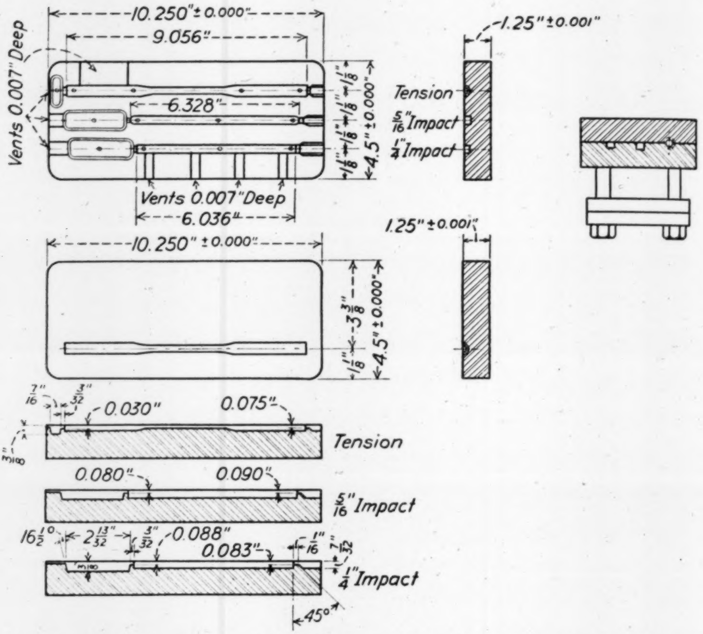
10.250" ± 0.000"
9.056"
Vents 0.007" Deep
6.328"
Vents 0.007" Deep
6.036"
1/8"
4.5" ± 0.000"
1.25" ± 0.001"
Tension
5/16" Impact
1/4" Impact
10.250" ± 0.000"
1.25" ± 0.001"
3 3/8"
1/8"
4.5" ± 0.000"
7/16"
3/32"
0.030"
0.075"
3/16"
Tension
0.080"
0.090"
5/16" Impact
16 1/2°
2 13/32"
3/32"
0.088"
0.083"
1/16"
7/32"
1/4" Impact
45°

FIG. 8.—Net Result of Changes Made to Die from its Original Construction Shown in Fig. 1.

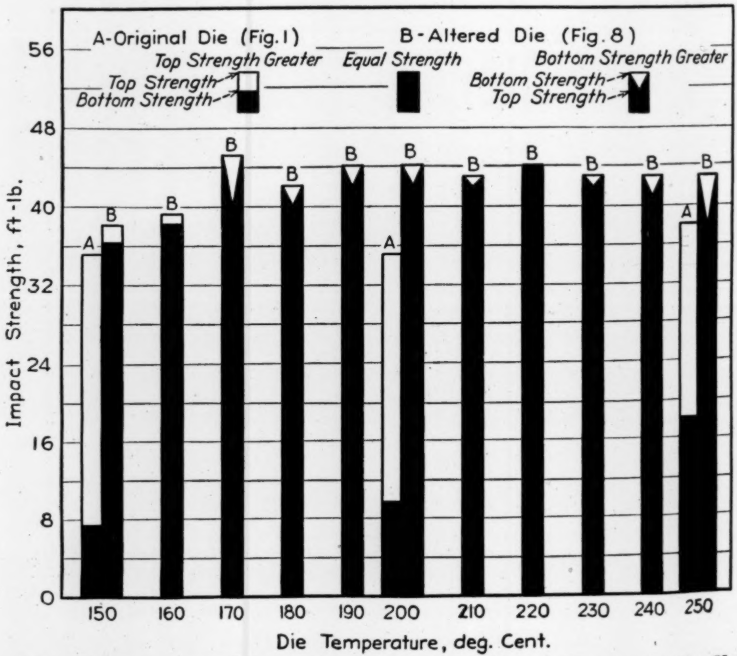

FIG. 9.—Total Improvement Effected in Impact Strength by Changes in the Unit Die.

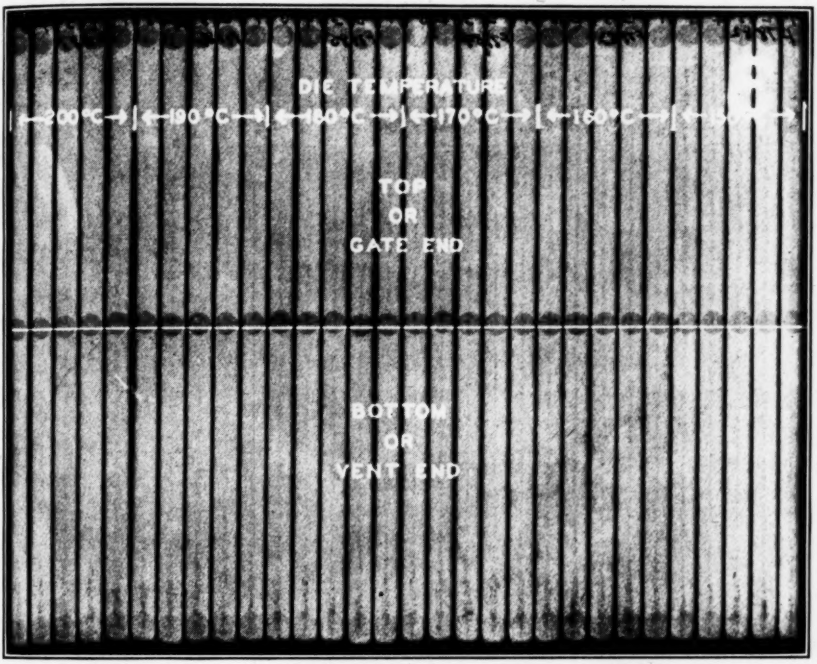
DIE TEMPERATURE
←200°C→
←190°C→
←180°C→
←170°C→
←160°C→
←150
TOP
OR
GATE END
BOTTOM
OR
VENT END

FIG. 10.—Radiograph of $\frac{1}{4}$-in. Impact Bars of Zamak 3 Cast at 150 to 200 C. in the Improved Unit Die.

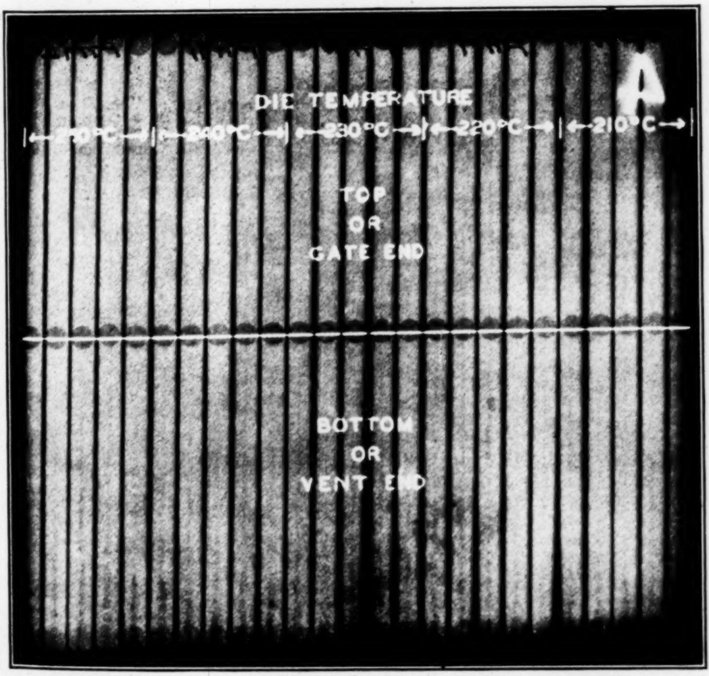
A
DIE TEMPERATURE
←250°C→
←240°C→
←230°C→
←220°C→
←210°C→
TOP
OR
GATE END
BOTTOM
OR
VENT END

FIG. 11.—Radiograph of $\frac{1}{4}$-in. Impact Bars of Zamak 3 Cast at 210 to 250 C. in the Improved Unit Die.

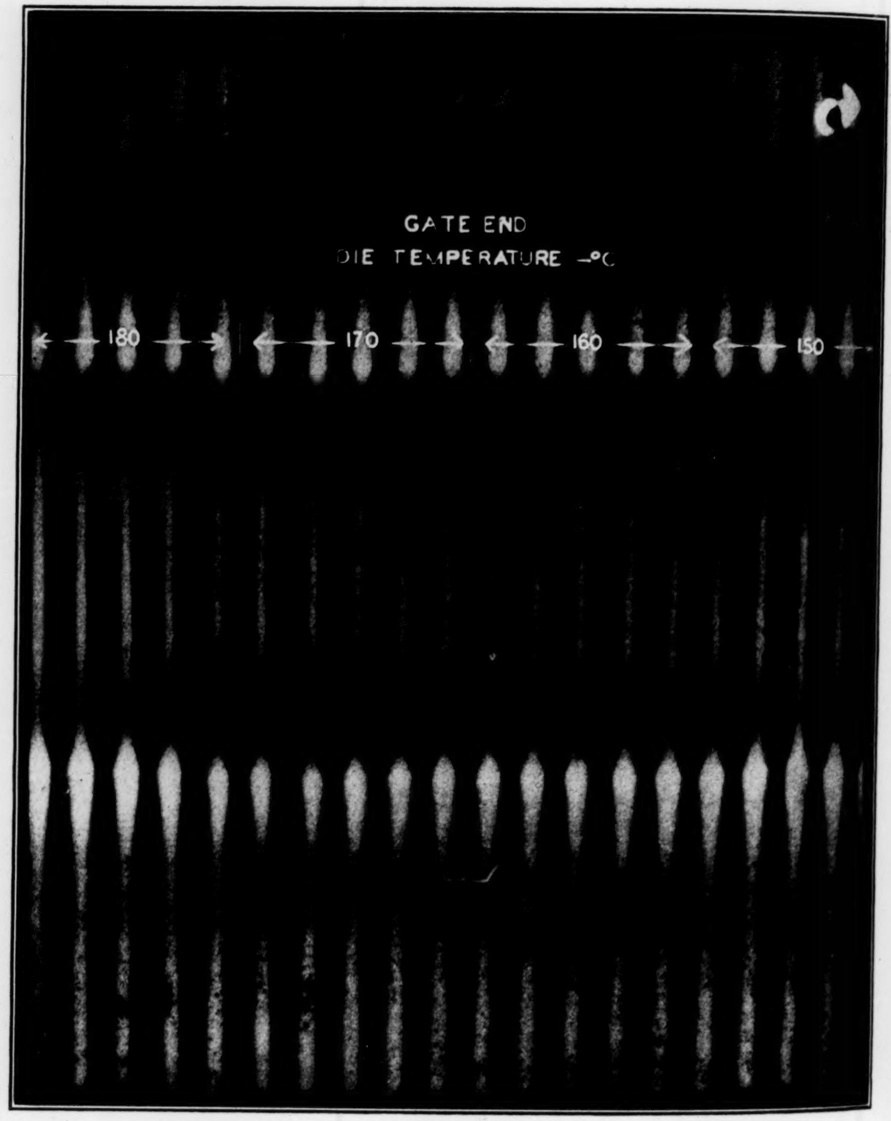
GATE END
DIE TEMPERATURE —°C
180
170
160
150

FIG. 12.—Radiograph of $\frac{1}{4}$-in. Round Tension Bars of Zamak 3 Cast at 150 to 180 C. in the Improved Unit Die.

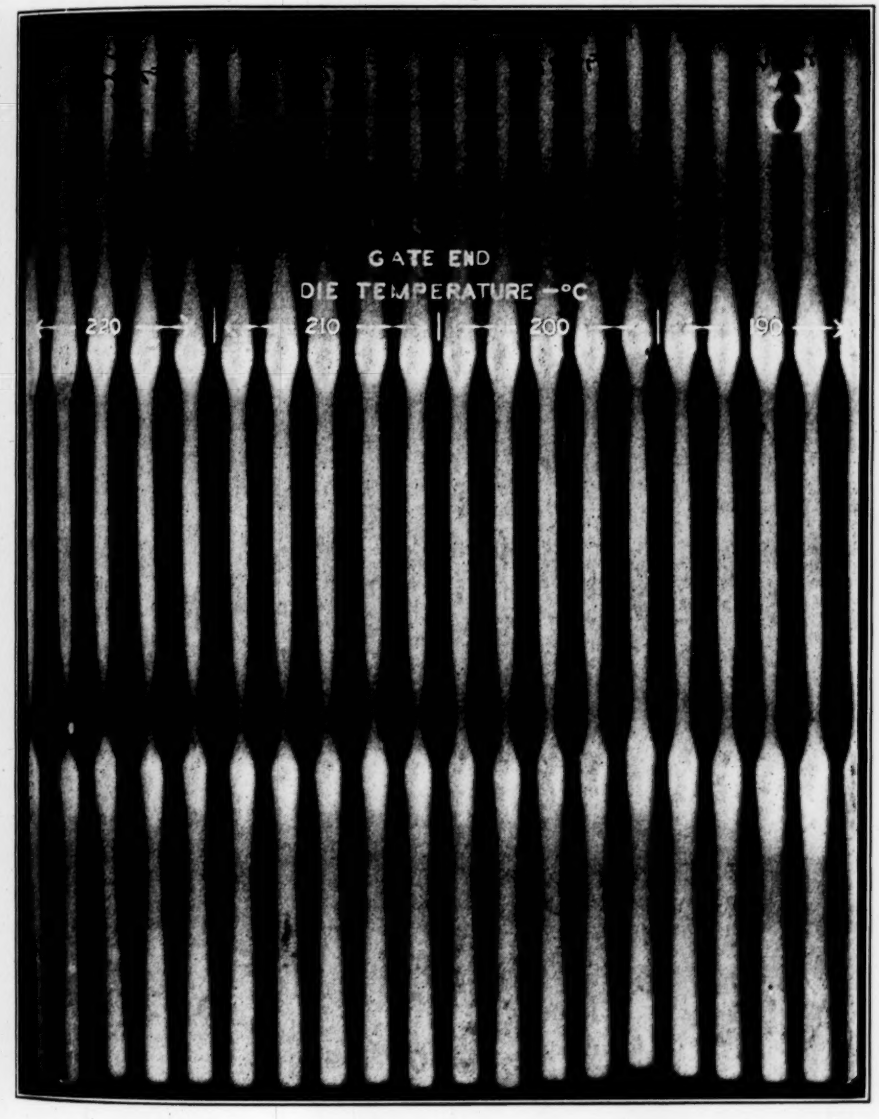
GATE END
DIE TEMPERATURE –°C
220
210
200
190

FIG. 13.—Radiograph of ¼-in. Round Tension Bars of Zamak 3 Cast at 190 to 220 C. in the Improved Unit Die.

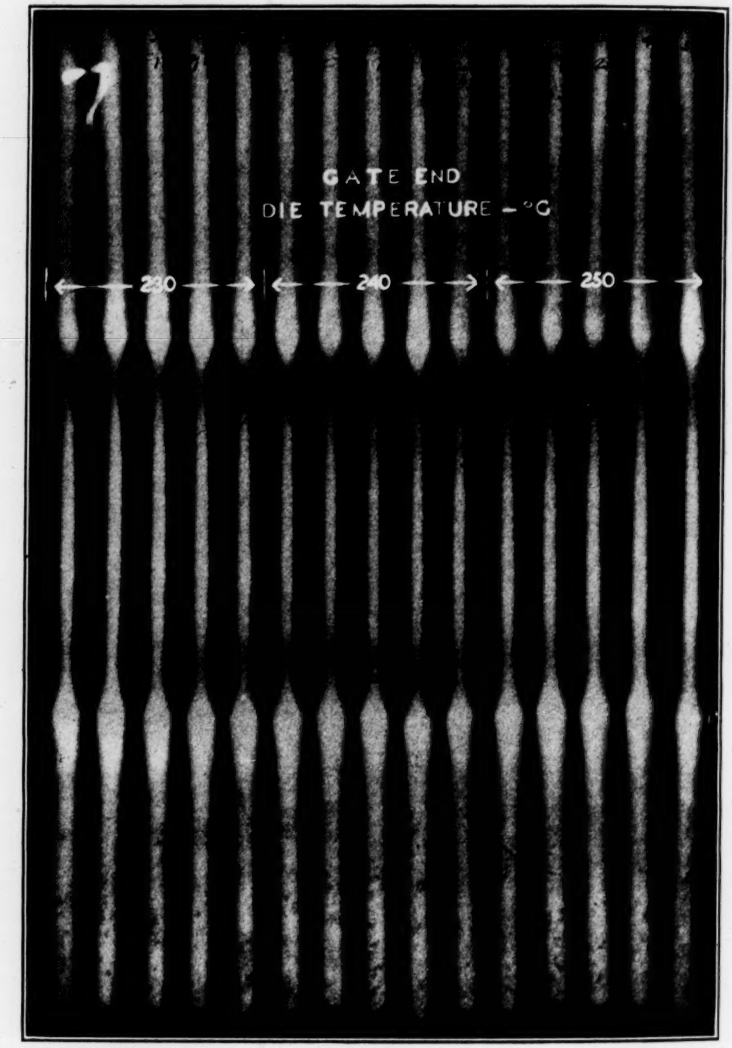
GATE END
DIE TEMPERATURE - °C
230
240
250

FIG. 14.—Radiograph of ¼-in. Round Tension Bars of Zamak 3 Cast at 230 to 250 C. in the Improved Unit Die.

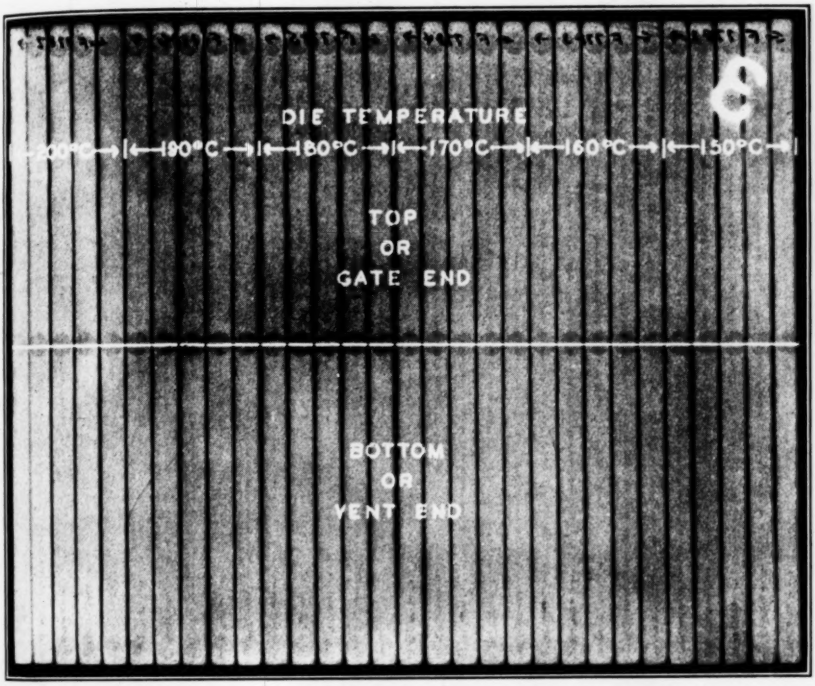
6
DIE TEMPERATURE
|←200°C→|←190°C→|←180°C→|←170°C→|←160°C→|←150°C→|
TOP
OR
GATE END
BOTTOM
OR
VENT END

FIG. 15.—Radiograph of $\frac{1}{4}$-in. Impact Bars of Zamak 5 Cast at 150 to 200 C. in the Improved Unit Die.

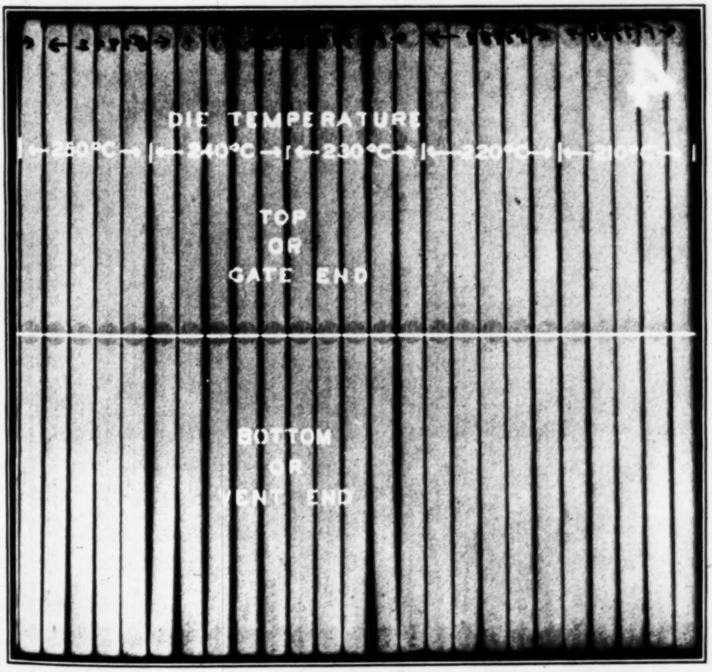
4
DIE TEMPERATURE
|←250°C→|←240°C→|←230°C→|←220°C→|←210°C→|
TOP
OR
GATE END
BOTTOM
OR
VENT END

FIG. 16.—Radiograph of $\frac{1}{4}$-in. Impact Bars of Zamak 5 Cast at 210 to 250 C. in the Improved Unit Die.

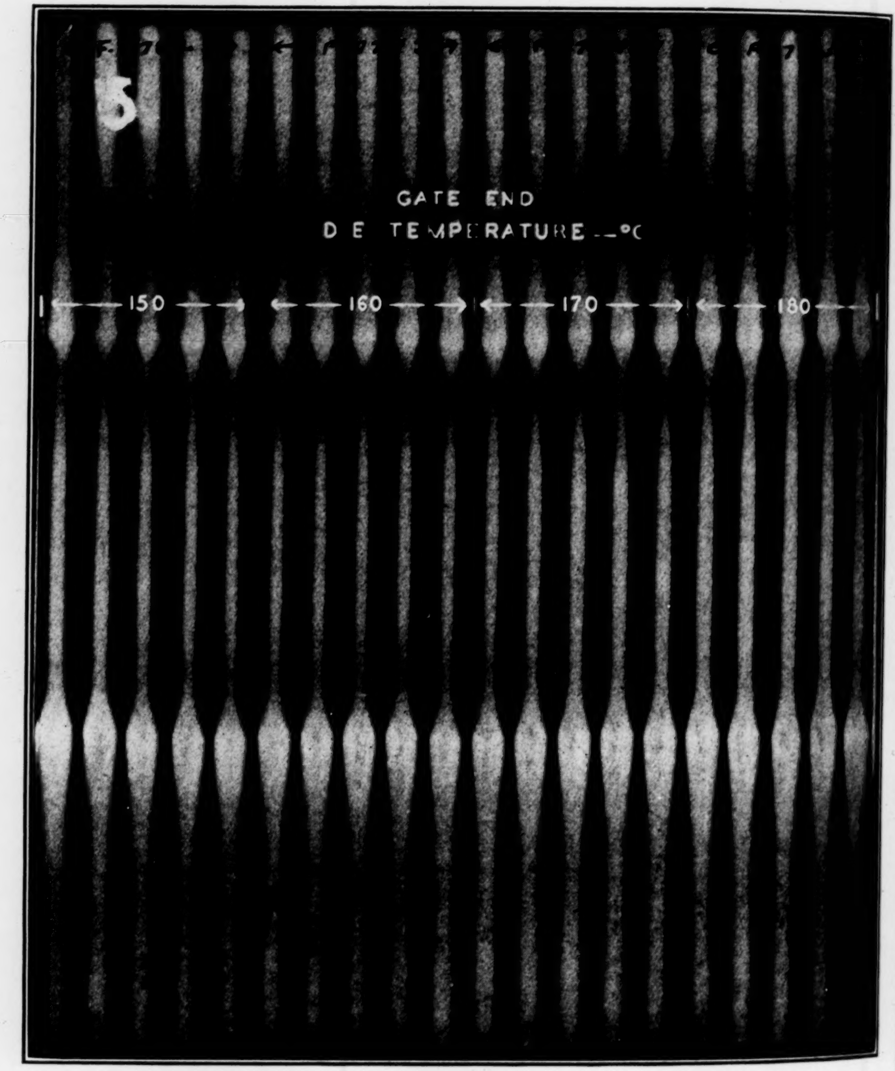
5
GATE END
DIE TEMPERATURE — °C
150
160
170
180

FIG. 17.—Radiograph of $\frac{1}{4}$-in. Round Tension Bars of Zamak 5 Cast at 150 to 180 C. in the Improved Unit Die.

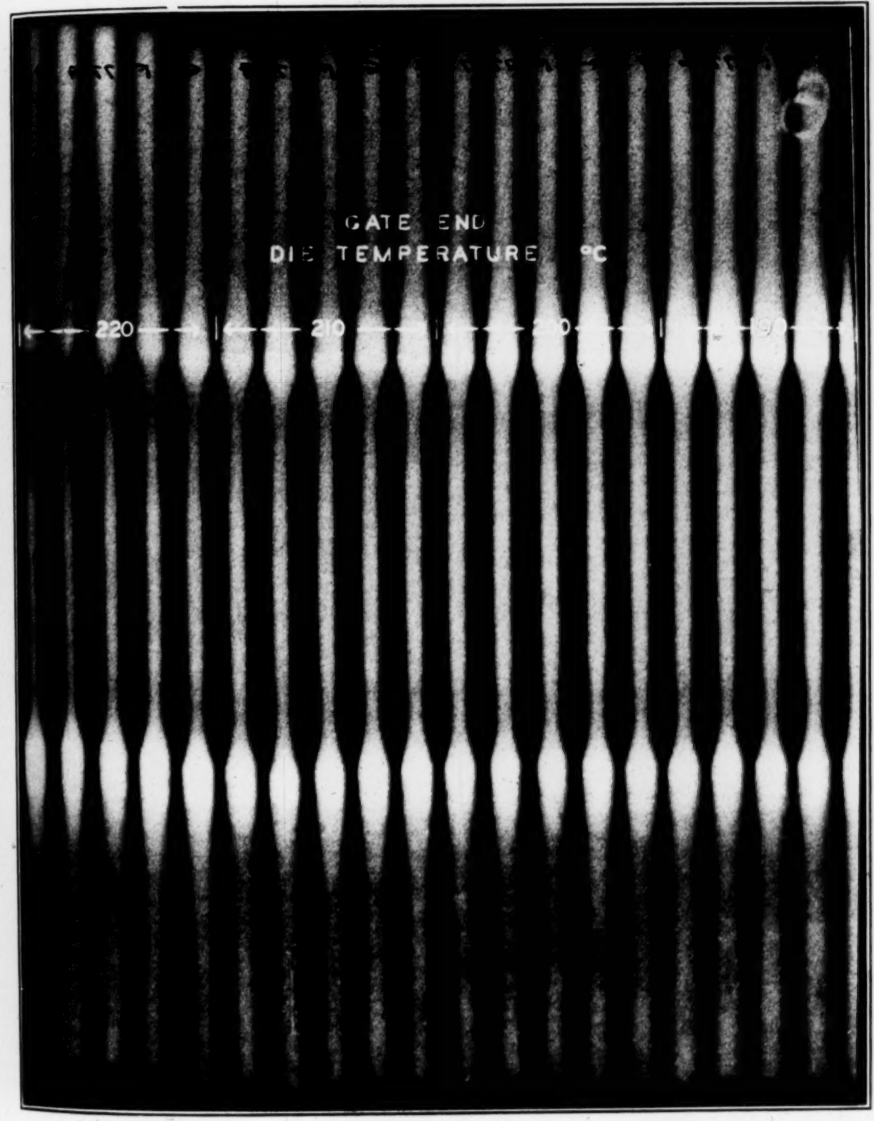
GATE END
DIE TEMPERATURE °C
220
210
200
190

FIG. 18.—Radiograph of $\frac{1}{4}$-in. Round Tension Bars of Zamak 5 Cast at 190 to 220 C. in the Improved Unit Die.

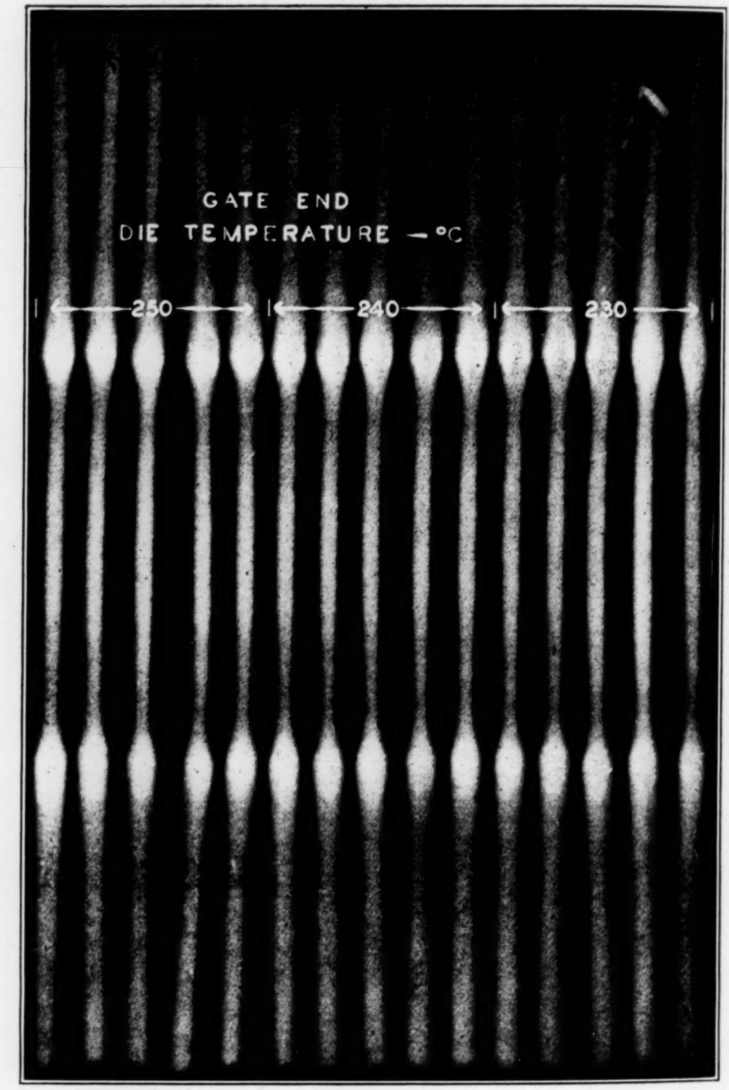
GATE END
DIE TEMPERATURE — °C
250
240
230

FIG. 19.—Radiograph of ¼-in. Round Tension Bars of Zamak 5 Cast at 230 to 250 C. in the Improved Unit Die.

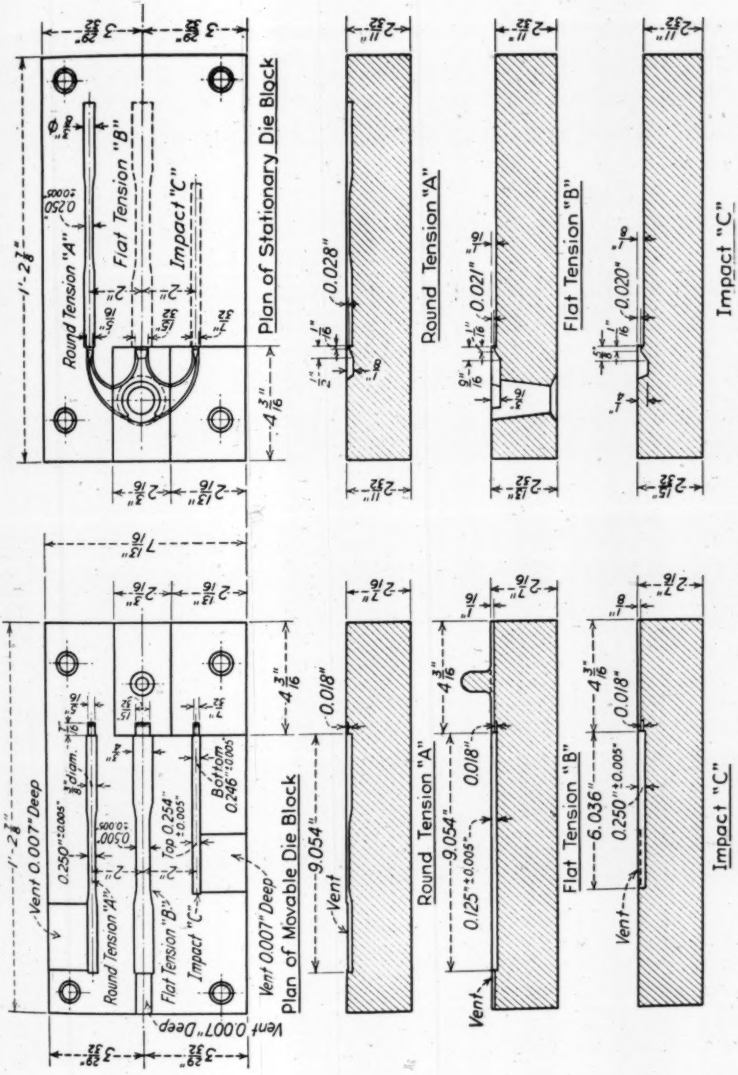
Plan of Stationary Die Block
Round Tension "A"
Flat Tension "B"
Impact "C"
1'-2 7/8"
4 3/16"
0.250 ±0.005
0.028"
0.021"
0.020"
Plan of Movable Die Block
Vent 0.007" Deep
7 13/16"
0.250"±0.005"
Top 0.254" ±0.005"
Bottom 0.246" ±0.005"
3/8" diam.
9.054"
6.036"
0.018"
0.125"±0.005"
0.250"±0.005"
Vent

FIG. 20.—Original Plan of A.S.T.M. Die for Production of ¼-in. Test Bars.

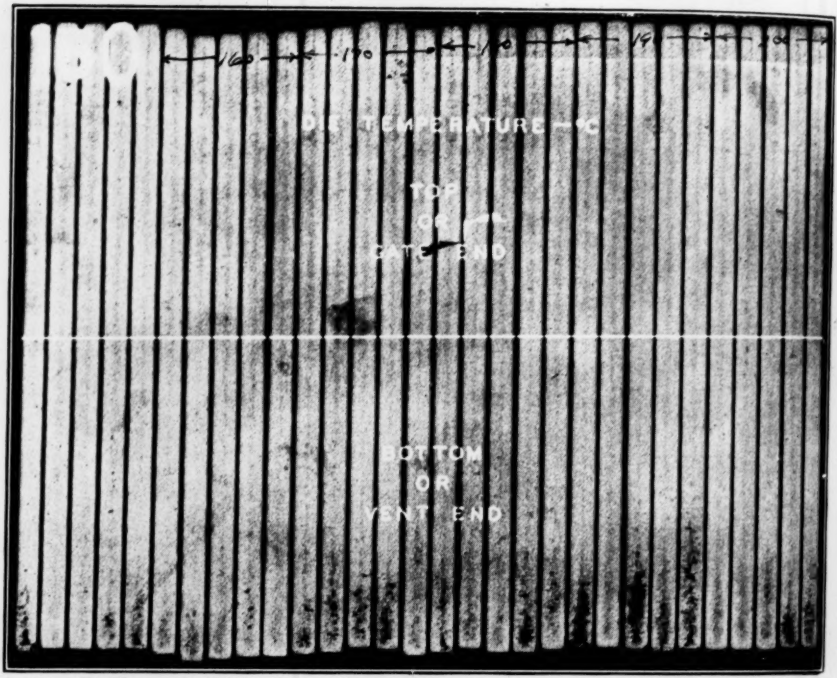
150
160
170
180
190
200
DIE TEMPERATURE—°C
TOP
OR
GATE END
BOTTOM
OR
VENT END

FIG. 21.—Radiograph of ¼-in. Impact Bars of Zamak 5 Cast at 150 to 200 C. in the A.S.T.M. Die.

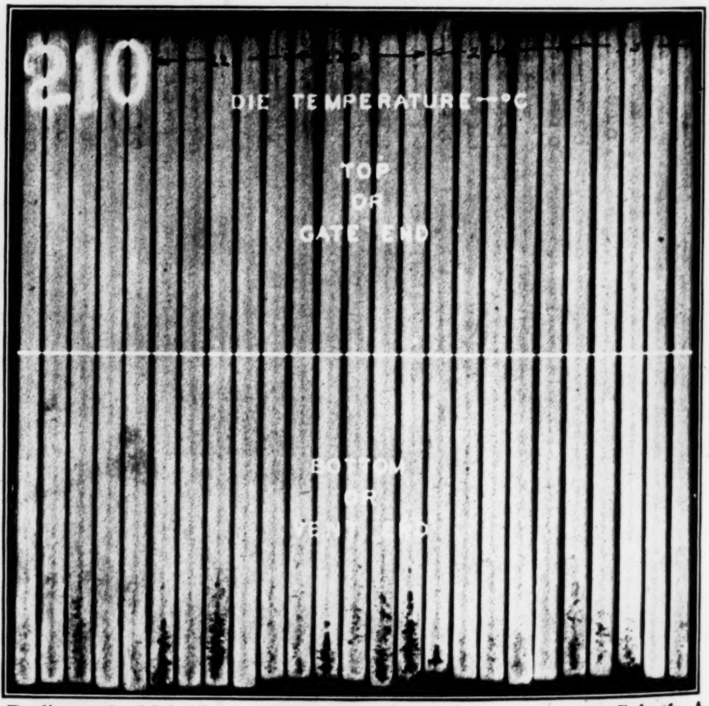
210
DIE TEMPERATURE—°C
TOP
OR
GATE END
BOTTOM
OR
VENT END

FIG. 22.—Radiograph of ¼-in. Impact Bars of Zamak 5 Cast at 210 to 250 C. in the A.S.T.M. Die.

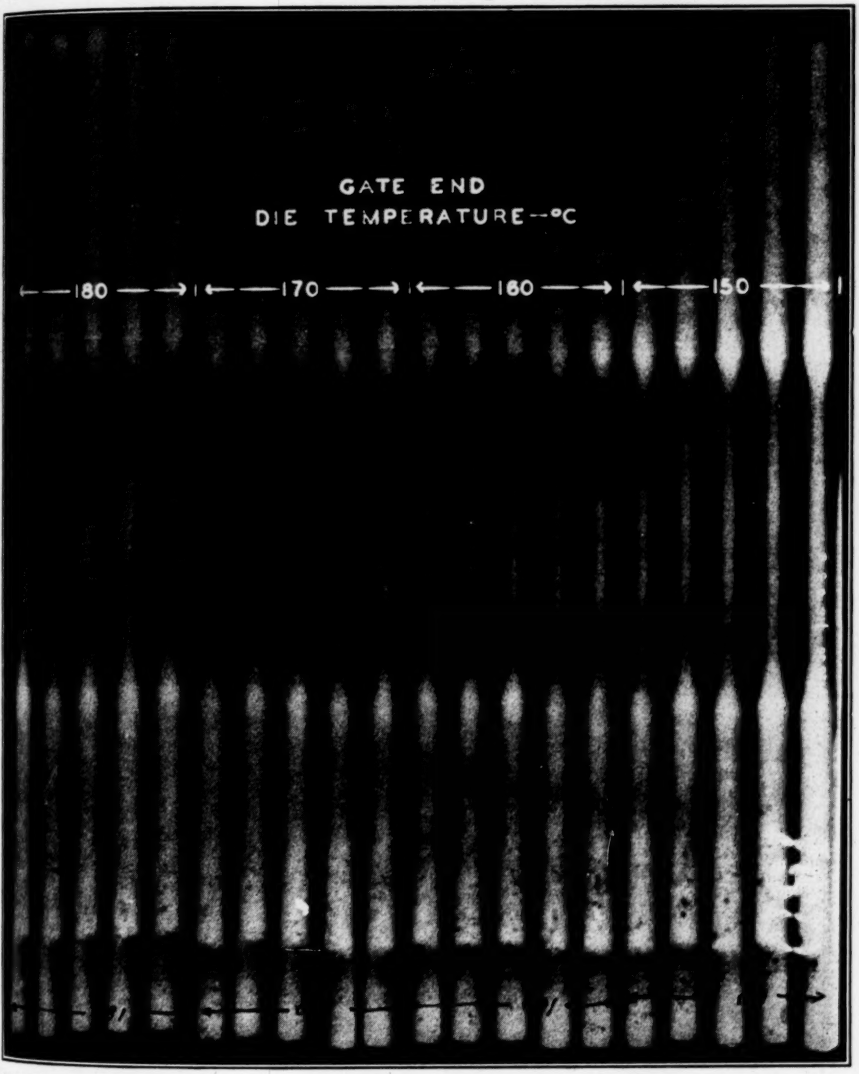
GATE END
DIE TEMPERATURE--°C
180
170
160
150

FIG. 23.—Radiograph of ½-in. Round Tension Bars of Zamak 5 Cast at 150 to 180 C. in the A.S.T.M. Die.

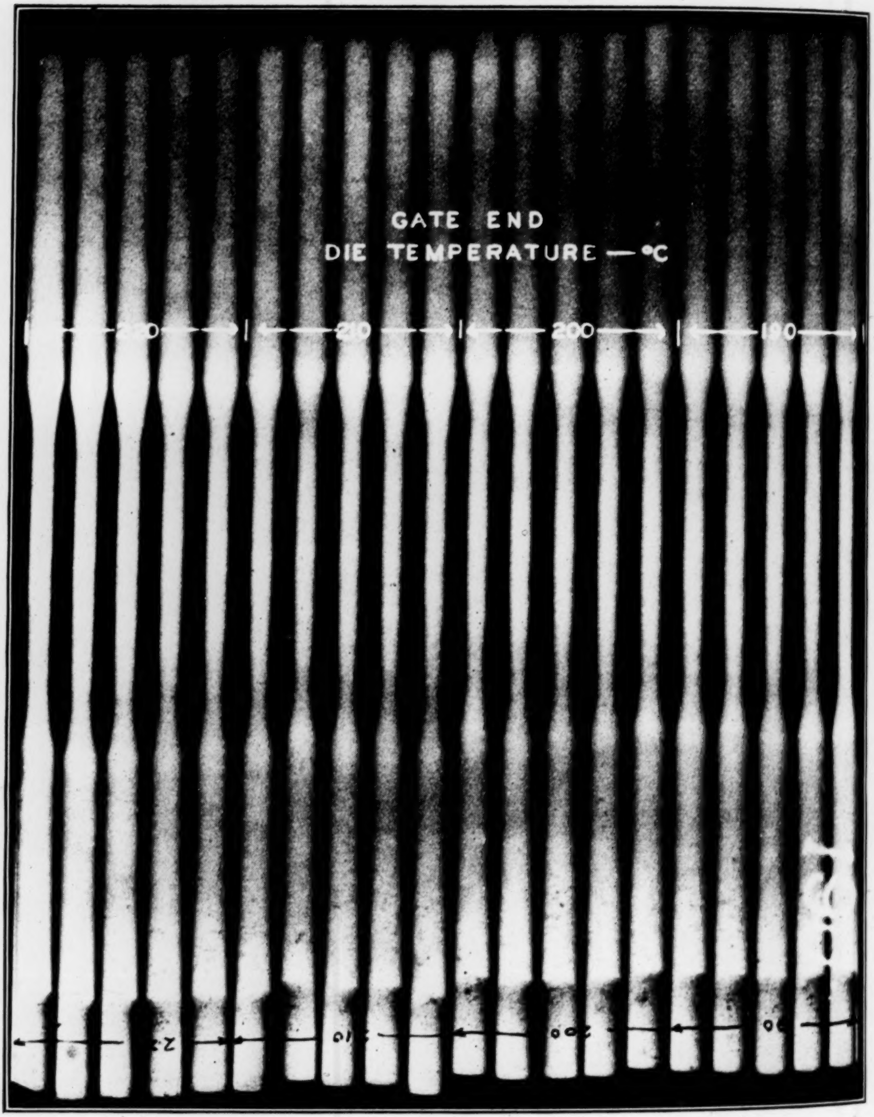
GATE END
DIE TEMPERATURE — °C
220
210
200
190

FIG. 24.—Radiograph of ¼-in. Round Tension Bars of Zamak 5 Cast at 190 to 220 C. in the A.S.T.M. Die.

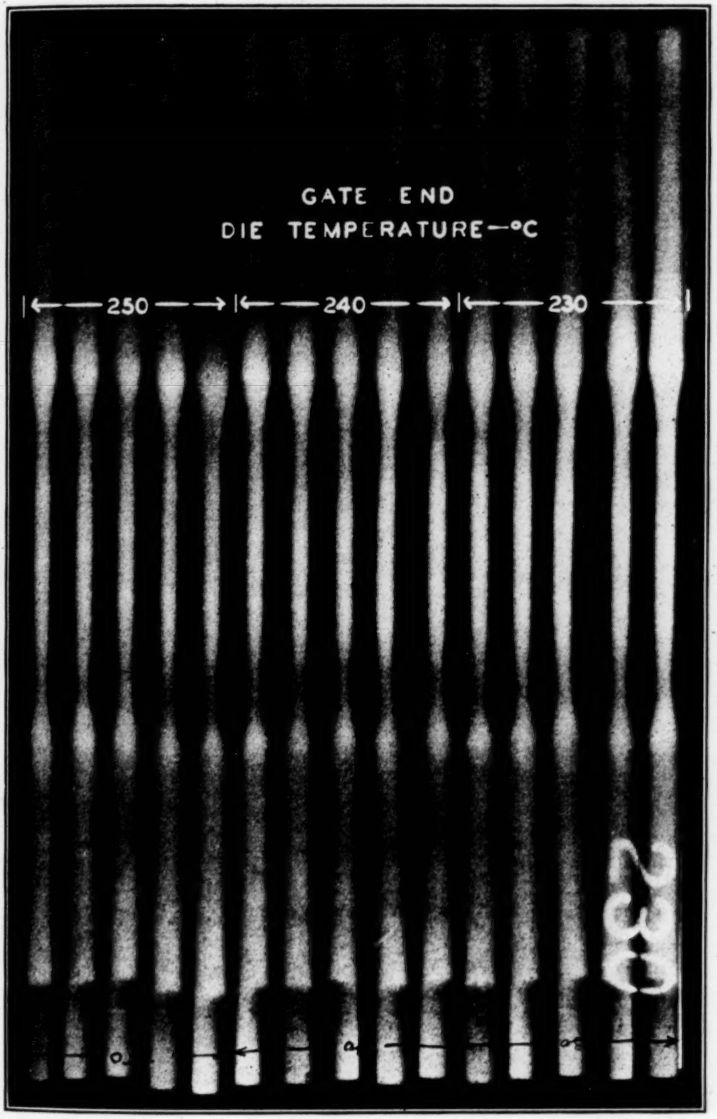
GATE END
DIE TEMPERATURE—°C
250
240
230
230

FIG. 25.—Radiograph of ¼-in. Round Tension Bars of Zamak 5 Cast at 230 to 250 C. in the A.S.T.M. Die.

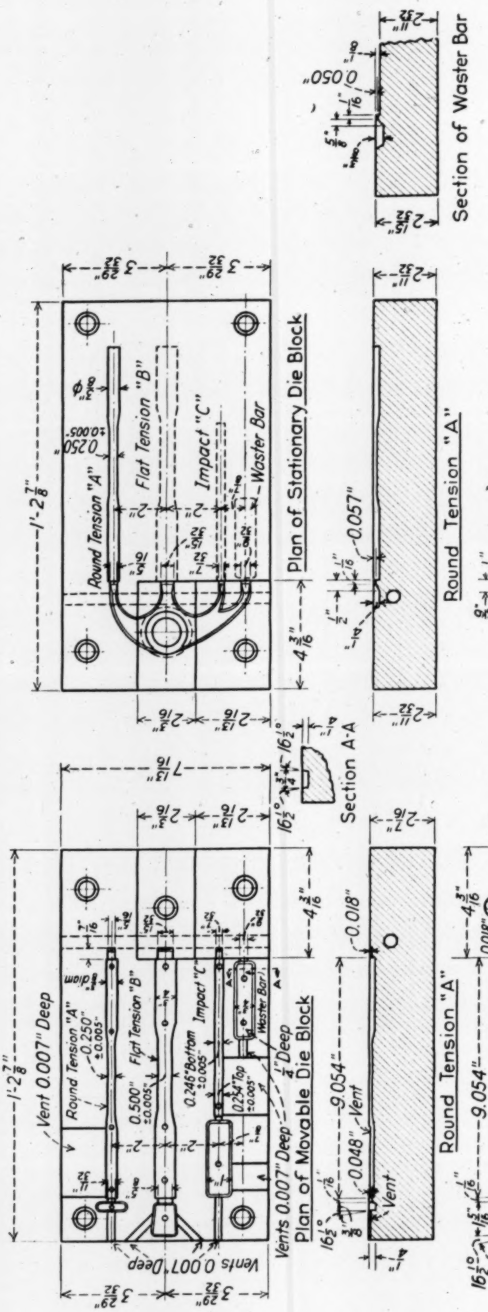
Section of Waster Bar
0.050"
Plan of Stationary Die Block
Round Tension "A"
Flat Tension "B"
Impact "C"
Waster Bar
1'-2 7/8"
3 29/32"
4 3/16"
0.250" ±0.005"
0.057"
0.042"
0.052"
Section A-A
16½°
Plan of Movable Die Block
Vent 0.007" Deep
Vents 0.007" Deep
1/4" Deep
0.500" ±0.005"
0.246" Bottom ±0.005"
0.234" Top ±0.005"
7 13/16"
2 13/16"
2 7/16"
0.018"
0.048"
9.054"
Vent
Water Cooled
0.125" ±0.005"
6.036"
0.070"
2 29/32"
2 11/32"
2 15/32"

FIG. 26.—Net Result of Changes Made to the Die from Its Original Construction Shown in Fig. 20.

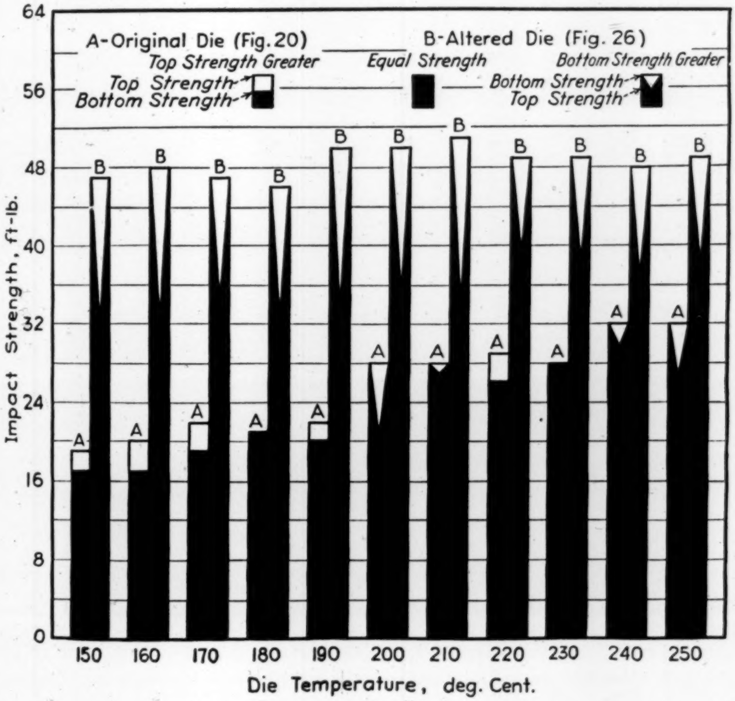

FIG. 27.—Total Improvement Effected in Impact Strength by Changes in the A.S.T.M. Die.

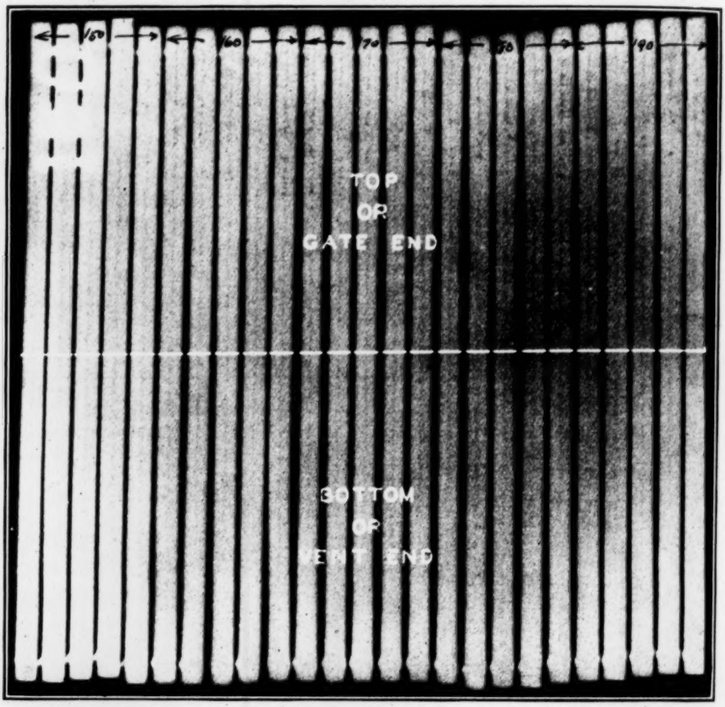
150
160
70
80
190
TOP
OR
GATE END
BOTTOM
OR
VENT END

FIG. 28.—Radiograph of $\frac{1}{4}$-in. Impact Bars of Zamak 5 Cast at 150 to 190 C. in the Improved A.S.T.M. Die.

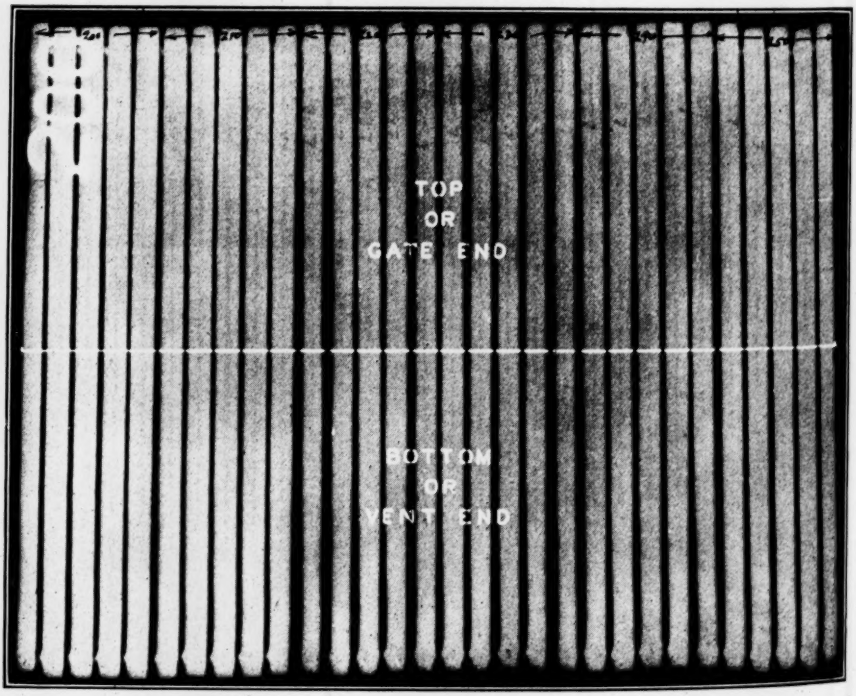
200
210
220
240
250
TOP
OR
GATE END
BOTTOM
OR
VENT END

FIG. 29. Radiograph of $\frac{1}{4}$-in. Impact Bars of Zamak 5 Cast at 200 to 250 C. in the Improved A.S.T.M. Die.

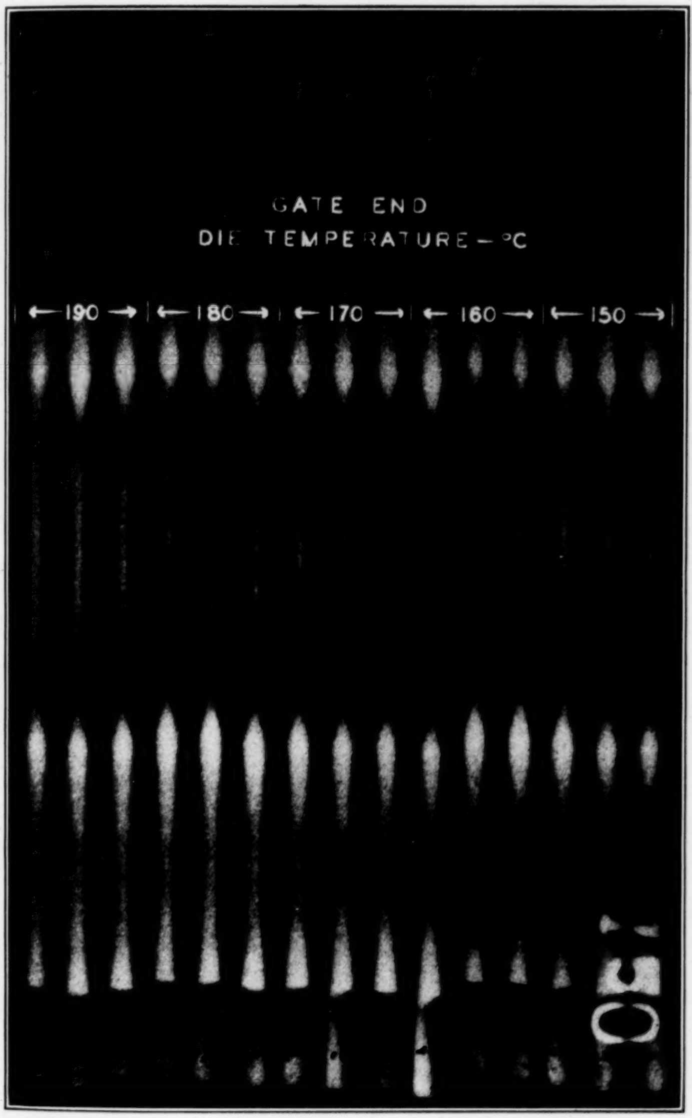
GATE END
DIE TEMPERATURE — °C
190
180
170
160
150
150

FIG. 30.—Radiograph of ½-in. Round Tension Bars of Zamak 5 Cast at 150 to 190 C. in the Improved A.S.T.M. Die.

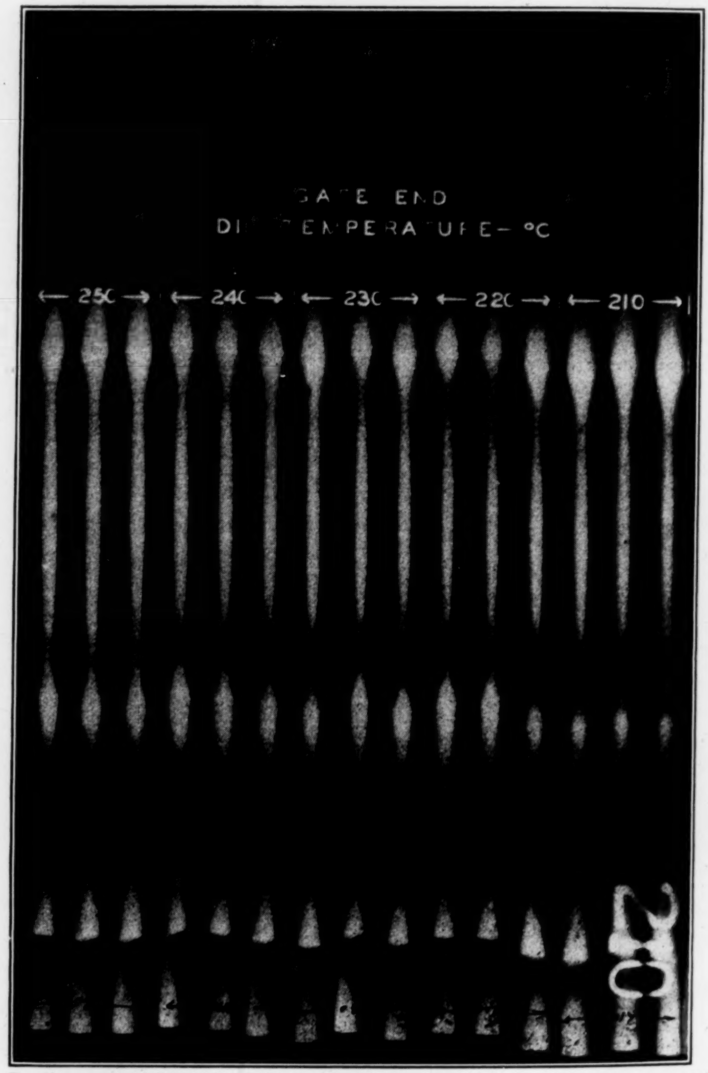
GATE END
DIE TEMPERATURE – °C
← 250 →
← 240 →
← 230 →
← 220 →
← 210 →

FIG. 31.—Radiograph of ¼-in. Round Tension Bars of Zamak 5 Cast at 210 to 250 C. in the Improved A.S.T.M. Die.

REPORT OF COMMITTEE B-7

ON

LIGHT METALS AND ALLOYS, CAST AND WROUGHT

Committee B-7 on Light Metals and Alloys, Cast and Wrought, has held two meetings during the year, at Atlantic City, N. J., on July 2, 1936, and at Chicago, Ill., on March 4, 1937.

Since the 1936 annual meeting the committee has lost two members by resignation and has gained two new members. The membership of the committee now totals 41, of whom 15 are classified as producer, 20 as consumer, and 6 as general interest members.

Subsequent to the 1936 annual meeting, Committee B-7 presented to the Society through Committee E-10 on Standards proposed Tentative Specifications for Aluminum-Base Alloy Permanent Mold Castings (B 108 - 36 T) and a proposed revision of the Tentative Specifications for Aluminum-Base Alloy Sand Castings (B 26 - 33 T). These recommendations were accepted[1] by Committee E-10 at a meeting held on August 26, 1936, and the specifications appear in the 1936 *Proceedings*.[2]

Later in the year Committee B-7 presented the following recommendations to the Society through Committee E-10:

Tentative Specifications for Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 - 36 T)

Revision of Tentative Specifications for:

Aluminum Sheet and Plate (B 25 - 34 T),
Aluminum-Alloy (Duralumin) Sheet and Plate (Aluminum-Copper-Magnesium-Manganese) (B 78 - 33 T),
Aluminum-Manganese Alloy Sheet and Plate (B 79 - 34 T), and
Aluminum-Alloy (Duralumin) Bars, Rods and Shapes (Aluminum-Copper-Magnesium-Manganese) (B 89 - 33 T).

These new and revised specifications were accepted[3] by Committee E-10 on November 30, 1936, and appear in the 1936 *Proceedings*.[4]

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee B-7 reported the following results of the letter ballot vote of a total of 34 ballots returned from a committee membership of 41: Specifications B 108, affirmative 27, negative 0, ballots marked "not voting" 7; Specifications B 26, affirmative 28, negative 0, ballots marked "not voting" 6.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 669, 674 (1936); also 1936 Book of A.S.T.M. Tentative Standards, pp. 238, 246.

[3] In submitting these recommendations to Committee E-10 on Standards, Committee B-7 reported results of the letter ballot vote as follows: Of a total membership of 41, 23 members returned their ballots, of whom 18 voted affirmatively, 0 negatively, and 5 members marked their ballots "not voting."

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 680 to 699 (1936); also 1936 Book of A.S.T.M. Tentative Standards, pp. 252 to 266.

RECOMMENDATIONS AFFECTING STANDARDS

I. Proposed Tentative Standard.—The committee recommends that the proposed Tentative Specifications for Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings, as appended hereto,[1,2] be accepted for publication as tentative. These specifications have been prepared in response to a request from the light metals industry.

II. Proposed Revision of Tentative Standard.—The committee recommends that the Tentative Specifications for Aluminum-Base Sand-Casting Alloys in Ingot Form (B 58 – 33 T)[3] be revised as appended hereto[1,4] and continued as tentative. These revisions have been made in order to bring the specifications into agreement with present commercial practice.

The committee also recommends that the Tentative Specifications for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 36 T)[5] and the Tentative Specifications for Aluminum-Base Alloy Sand Castings (B 26 – 36 T)[5] be revised by the deletion of Table II which includes precision requirements that were originally intended to be applicable in the chemical analysis of these alloys. The specifications in their revised form are appended hereto.[6]

These recommendations have been submitted to letter ballot of the committee which consists of 41 members, the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. PROPOSED TENTATIVE STANDARD			
Specifications for Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings	28	0	5
II. PROPOSED REVISION OF TENTATIVE STANDARDS			
Specifications for Aluminum-Base Sand-Casting Alloys in Ingot Form (B 58 - 33 T)	27	0	6
Specifications for Aluminum-Base Alloy Permanent Mold Castings (B 108 - 36 T)	29	0	4
Specifications for Aluminum-Base Alloy Sand Castings (B 26 - 36 T)	28	0	5

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum and Aluminum Alloy Ingots (D. L. Colwell, chairman).—This subcommittee has prepared proposed Tentative Specifications for Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings and has completed the revision of the Tentative Specifications for Aluminum-Base Sand-Casting Alloys in Ingot Form (B 58 – 33 T),

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[1] Revisions of these specifications submitted to the Society at the annual meeting, see Summary of Proceedings, p. 29, have been incorporated in the specifications.

[2] See p. 671.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 592 (1933); also 1936 Book of A.S.T.M. Tentative Standards, p. 243.

[4] See p. 668.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 669, 674 (1936); also 1936 Book of A.S.T.M. Tentative Standards, pp. 238, 246.

[6] See pp. 674 and 678.

as mentioned earlier in this report. The subcommittee also has in preparation specifications for aluminum-base alloys in ingot form for die castings. This work will be done in cooperation with Committee B-6 on Die-Cast Metals and Alloys in order that the ingot specifications will be consistent with the product specifications as covered by the Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 – 33 T).

Subcommittee V on Testing Light Metals (R. L. Templin, chairman).—This subcommittee has given considerable study to various problems relating to the testing of light metals. Special attention has been given to the modulus of elasticity of sand cast alloys and the data obtained appear as Appendix I to this report. Other problems being considered by this subcommittee include hardness and the effect of speed of testing and foundry technique on the physical properties of light-metal alloy test bars. This letter problem includes a study of the effects of test bar design, gating, and chilling,

Subcommittee VI on Anodic Oxidation of Aluminum and Aluminum Alloys (T. S. Fuller, chairman).—One subgroup of this subcommittee, under the leadership of Mr. D. Basch, has circularized various industries using aluminum and its alloys in order to determine the requirements of the trade for anodized coatings. These data are being summarized and will be used as a guide in the study of methods of testing anodized coatings.

Another subgroup under the guidance of Mr. J. D. Edwards has accumulated data and information on methods of testing oxide coatings on aluminum which appears as Appendix II to this report.

This report has been submitted to letter ballot of the committee which consists of 41 members; 34 members returned their ballots, of whom 33 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

SAM TOUR,
Chairman.

J. A. GANN,
Secretary.

APPENDIX I

MODULUS OF ELASTICITY OF ALUMINUM ALLOYS

By R. L. Templin[1] and Sam Tour[2]

At the request of Subcommittee V on Testing Light Metals, of Committee B-7, careful stress-strain tests have been made for the purpose of defining moduli of elasticity for the aluminum-base alloys covered by A.S.T.M. product specifications. There are at the present time the following tentative specifications of the Society, covering aluminum-base alloys:

Tentative Specifications for:

B 25 – 36 T.	Aluminum Sheet and Plate[3]
B 79 – 36 T.	Aluminum-Manganese Alloy Sheet and Plate[3]
B 109 – 36 T.	Aluminum-Magnesium-Chromium Alloy Sheet and Plate[4]
B 78 – 36 T.	Aluminum-Alloy (Duralumin) Sheet and Plate (Aluminum-Copper-Magnesium-Manganese)[3]
B 89 – 36 T.	Aluminum-Alloy (Duralumin) Bars, Rods and Shapes (Aluminum-Copper-Magnesium-Manganese)[3]
B 26 – 36 T.	Aluminum-Base Alloy Sand Castings[3]
B 108 – 36 T.	Aluminum-Base Alloy Permanent Mold Castings[3]
B 85 – 33 T.	Aluminum-Base Alloy Die Castings[5]

As a result of a large number of tests made over a period of years, a modulus of elasticity value of 10,000,000 lb. per sq. in. has been quite generally accepted for commercial aluminum (B 25 – 36 T) and for the alloy containing 1.0 to 1.5 per cent magnesium (B 79 – 36 T); and for the so-called strong aluminum alloys, containing greater amounts of alloying constituents, 10,300,000 lb. per sq. in. has had general acceptance. These latter alloys include the aluminum-magnesium-chromium alloy covered by Specifications B 109 – 36 T and duralumin (B 78 – 36 T and B 89 – 36 T). In the case of the cast alloys, however, there has not been such general agreement and for this reason Subcommittee V requested further information on these alloys.

In order to provide as reliable data as possible, bars 1 in. in diameter and 14 in. in length were cast and each bar was examined by X-ray to insure soundness. In determining modulus of elasticity it is extremely important that the specimens used be uniform, sound, and free from defects. Specimens of the following alloys were prepared:

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[1] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

[2] Vice-President, Lucius Pitkin, Inc., New York City.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 685, 694, 690, 680, 674 and 669, respectively (1936); also 1936 Book of A.S.T.M. Tentative Standards, pp. 257, 266, 262, 252, 246 and 238, respectively.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 699 (1936).

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 641 (1933); also 1936 Book of A.S.T.M. Tentative Standards, p. 348.

MARKED	ALCOA ALLOY	A.S.T.M. ALLOY DESIGNATION
L-1173	No. 112	C
L-1178	No. 212	CC
L-1176	No. 122	F
L-1168	No. 195–T6	G[a]
L-1171	No. 142–T61	H
L-1174	No. 43	J
L-1175	No. 47	K
L-1177	No. 214	L
L-1170	No. 356–T6	M[a]
L-1169	No. 355–T6	N[a]

[a] Heat treatment No. 2.

No specimens of A.S.T.M. alloys GG and JJ were tested because the differences in composition between these and alloys G and J were considered insufficient to affect the values of modulus of elasticity. For a similar reason it was considered unnecessary to test the material in more than one condition of heat treatment.

The chemical analyses of these bars are shown in Table I, as are also the results of tension tests. These tests were made on standard ½-in. diameter test specimens cast from the same heats of metal as the specimen for the modulus of elasticity tests. The chemical compositions, tensile strengths and elongations all meet the requirements of Specifications B 26 – 36 T.

TABLE I.—CHEMICAL COMPOSITION AND TENSILE PROPERTIES OF ALLOYS.

Alcoa Alloy	A.S.T.M. Alloy Designation	Chemical Composition, per cent						Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent
		Copper	Iron	Silicon	Magnesium	Nickel	Zinc		
No. 112	C	7.45	1.02	2.13	....	....	1.99	22 400	1.0
No. 212	CC	7.89	0.81	1.14	....	....	....	23 100	2.5
No. 122	F	9.81	1.18	0.54	0.26	....	....	27 200	0.0
No. 195–T6	G	4.30	0.55	0.76	....	....	....	35 400	5.0
No. 142–T61	H	4.12	0.35	0.17	1.17	2.18	....	35 200	0.0
No. 43	J	0.07	0.66	5.39	....	....	....	19 500	5.0
No. 47	K	0.01	0.45	12.39	....	....	....	25 300	11.0
No. 214	L	0.02	0.13	0.07	3.98	....	....	26 300	13.5
No. 356–T6	M	0.015	0.23	6.98	0.36	....	....	30 000	3.0
No. 355–T6	N	1.18	0.32	4.86	0.53	0.03	....	33 500	2.0

Duplicate tests were made on each alloy. The specimen used for the modulus of elasticity determinations was similar to the A.S.T.M. ½-in. diameter specimen with threaded ends, except that the reduced section was 10 in. in length. The reduced section was uniform in diameter throughout its length.

In the stress-strain tests the loads were applied using a 20,000-lb. capacity Amsler hydraulic testing machine and the strains were measured over an 8-in. gage length by means of a Ewing extensometer, each of which had previously been carefully calibrated and found to be correct within ±1 per cent. In each test, increments of stress were applied and the corresponding strains were measured until the proportional limit of the material was exceeded. The modulus of elasticity values were determined by plotting differences between observed strains and strains calculated from a trial modulus, following the method described by L. B. Tuckerman.[1]

The results of the tests made in this investigation are summarized in Table II. In these tests, which were made with extreme care, the modulus values obtained on duplicate specimens checked within close limits. The modulus of elasticity values range from 10,110,000 lb. per sq. in. (alloy L) to 10,860,000 lb. per sq. in. (alloy K). While the modulus of elasticity values for these alloys average about 10,390,000 lb. per sq. in., it will be noted that in only one case, that of alloy K, the modulus varies more than 3 per cent from the value generally accepted for wrought aluminum alloys, 10,300,000 lb. per sq. in. It seems reasonable, therefore, to accept for practical purposes a value of 10,300,000 lb. per sq. in. for the alloys covered by Specifications B 26 – 36 T.

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[1] Discussion by L. B. Tuckerman of paper by R. L. Templin, "The Determination and Significance of the Proportional Limit in the Testing of Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 538 (1929).

TABLE II.—MODULUS OF ELASTICITY.

Alcoa Alloy	A.S.T.M. Alloy Designation	Modulus of Elasticity, lb. per sq. in.			Variation from 10,300,000 lb. per sq. in., per cent
		Specimen 1	Specimen 2	Average	
No. 112	C	10 200 000	10 350 000	10 275 000	−0.2
No. 212	CC	10 220 000	10 430 000	10 325 000	+0.2
No. 122	F	10 540 000	10 600 000	10 570 000	+2.6
No. 195-T6	G	10 170 000	10 150 000	10 160 000	−1.4
No. 142-T61	H	10 420 000	10 420 000	10 420 000	+1.2
No. 43	J	10 450 000	10 370 000	10 410 000	+1.1
No. 47	K	10 800 000	10 920 000	10 860 000	+5.4
No. 214	L	10 200 000	10 020 000	10 110 000	−1.8
No. 356-T6	M	10 520 000	10 500 000	10 510 000	+2.0
No. 355-T6	N	10 330 000	10 280 000	10 305 000	0.0

No molds were available for making permanent-mold and die-cast specimens suitable for modulus of elasticity determinations and consequently no tests of these alloys were made. In the absence of such data it is recommended that a modulus of elasticity value of 10,300,000 lb. per sq. in. be used for the alloys covered by Specifications B 108 – 36 T and B 85 – 33 T.

APPENDIX II

REPORT ON METHODS OF TESTING OXIDE COATINGS ON ALUMINUM

During recent years, the serviceable characteristics of oxide coatings on aluminum have received general recognition. As a result, their applications have increased at a remarkable rate. The oxide coatings which have received most attention are those produced by making aluminum and its alloys anode in an electrolyte such as sulfuric acid, oxalic acid, chromic acid, or other electrolytes generally acid in nature. These coatings, generally speaking, are of substantial thickness and are useful because of their hardness and resistance to wear. Considerable attention is now being paid, also, to oxide films produced solely by chemical reaction. Thin anodic films of the type employed on electrolytic devices such as condensers, rectifiers, and lightning arresters, are not included in the present discussion.

With the extended use of oxide coatings on aluminum there has come a demand for testing methods suitable for appraising their service characteristics. In many ways these coatings are so different from electroplates, paint films and other non-metallic coatings, that special methods for testing have had to be devised. The interest in this subject led to the appointment by Committee B-7 of Subcommittee VI on Anodic Oxidation of Aluminum and Aluminum Alloys to consider the general subject of anodic coatings, and a section of this subcommittee was appointed to study testing methods. The work of this section, comprised of Messrs. H. G. Arlt, W. E. McCullough and J. D. Edwards, chairman, has resulted in the development of some information on methods of testing oxide coatings which is presented in this report. It is recognized, of course, that much of this work is only preliminary in nature, and it is hoped that the suggestions and criticisms of those interested in this field will be freely offered, so that the development of satisfactory methods will be facilitated.

Investigation of testing methods involved the collaboration of a number of laboratories. For the work of these cooperating laboratories, a supply of oxide-coated aluminum was produced in the plant of the Aluminum Company of America. Enough material of each type was produced so that the cooperating parties could work on substantially identical material and hence make their test results directly comparable. For these tests, materials of two different compositions were chosen: the first was commercial aluminum, known as 2S-H (the hard temper), and the second material was an alloy, 53S-T, containing small amounts of magnesium, silicon and chromium as the alloying constituents. The latter alloy was selected because it has been widely used with the anodic finish for architectural and other applications. These two materials in the form of large sheets (No. 18 gage) were anodically treated by the Alumilite process, using sulfuric acid electrolyte. The sheets were oxidized for four different periods, so as to give samples having four different coating thicknesses. The edges of the sheet near the current-carrying clamps were then sheared off and discarded and the remainder of the sheet was cut up into test samples.

THICKNESS OF OXIDE COATINGS

Experience in the use of oxide coatings has shown that the thickness of a coating may be an important measure of its suitability for some particular application. It may be an indication of the resistance to wear which may be expected, the resistance to corrosion, and perhaps other useful properties of the coating. Methods for the measurement of coating thickness are therefore of primary importance, both for investigational purposes and for routine control work.

Oxide coatings employed for their mechanical properties generally have a thickness in excess of 0.0001 in. and are less than 0.001 in. in thickness. This is in contrast to the anodic films used on electrolytic devices, where the oxide film has a thickness on the order of 0.00001 in. In passing, it may be noted that paint films are usually on the order of 0.001 in. in thickness per coat, although films both thinner and thicker than this value are in common use. Moreover, paint films, as generally used, consist of two or more coats.

The most exact determinations of oxide coating thickness are made by direct measurement under the microscope. The sample to be measured is mounted and polished as for metallographic examination, so that a cross-section of the film is presented for examination. The thickness of the coating is then determined by measurement with a calibrated micrometer eyepiece.

Measurement of Coating Thickness with the Microscope:

The method used in the Aluminum Research Laboratories consists in making up a composite specimen of a number of pieces of the coated sample. A spacer of aluminum sheet is placed between each sample of coated sheet. This spacer protects the oxide coating during the polishing operation. A composite specimen of this type is made of two or more pieces of the coated sample. Each piece should be $\frac{1}{2}$ to $\frac{3}{4}$ in. in length and $\frac{3}{8}$ to $\frac{1}{2}$ in. in width. The pieces can be clamped together to form a pack by drilling two holes and using machine screws to hold the assembly together. The material used for spacers between the coated sheets should be annealed aluminum sheet if the surface of the oxidized sample is rough, or hard-rolled aluminum sheet can be used and is preferred if the surface of the oxidized specimen is smooth. Special precaution should be taken to avoid excessive pressure in clamping the composite specimen, otherwise the coating will crack. In making metallographic specimens of samples from oxide-coated castings, it is desirable to place a backing-up strip of aluminum sheet against the coated surface before mounting the specimen. Specimens from cast samples can be mounted in sulfur, Bakelite, Vinylite, Tenite, or other suitable plastic material, if molding equipment is available.

After a composite sample of oxide-coated sheet or cast products has been made, it can be polished with the same procedure as other aluminum specimens. Because the oxide coating is both hard and friable, special care must be taken in making up a specimen in order to prevent rounding off the edges or fracturing of the coating during polishing. The method commonly employed for polishing aluminum specimens consists of the following operations:

(*a*) First, a plane surface is obtained by rubbing the specimen on a mill file and on Nos. 0, 00, and 000 metallographic emery paper in the order named.

(*b*) Then the specimen should be polished on a rotating disk operating at about 300 r.p.m., using a kitten's ear broadcloth pad and No. 600 alundum flour.

(*c*) The final polishing operation is carried out on a rotating disk operating at about 150 r.p.m. In this operation, heavy magnesium oxide powder is used on a kitten's ear broadcloth pad moistened with distilled water.

After a specimen has been properly polished, it should be washed in warm water and blown dry. The polished surface should not be touched or rubbed against anything, otherwise the finish will be spoiled. In general, the examination of oxide coatings on aluminum specimens is carried out on unetched specimens. Etching with the reagents commonly employed for aluminum alloys tends to pit and dissolve the oxide coating. Since the optical properties of oxide coatings are quite different from those of aluminum, it is not necessary to employ an etching reagent in order to differentiate between the oxide coating and the metal.

Measurement of Coating Thickness by Stripping Method:

An approximate measurement of film thickness suitable for routine control can be made by measuring the thickness of the coated piece with a micrometer caliper before and after removal of the oxide coating by chemical means. The problem is to find a reagent which dissolves the oxide coating with suitable rapidity but which attacks the aluminum base only slowly, if at all. A method operating along these lines has been developed in the Aluminum Research Laboratories. In operation, a small specimen of the coated metal is immersed in a hot sulfuric acid solution containing antimony. The coating is quickly dissolved and when the bare aluminum has been exposed, antimony deposits as a gray to black film and indicates that the stripping operation is complete. Details of the method are as follows:

Apparatus.—(*a*) A sharp pointed steel wire.

(*b*) Micrometer caliper, Brown & Sharpe Catalog No. 233, with both anvil and spindle conically pointed and flattened to $\frac{1}{32}$ in. in diameter.

Reagents.—A $\frac{7}{8}$ N H_2SO_4 solution containing 2 g. per liter of antimony trioxide. The acid functions as a stripping solution and the antimony functions as an indicator of completeness of stripping by deposition of antimony when bare metal is exposed.

Procedure.—(*a*) Before making the stripping test, a qualitative test should be made to determine if the coating is sealed or unsealed and, if possible, the type of alloy. For the preliminary test, bring the stripping mixture to a boil in a Pyrex beaker, carefully noting the volume, immerse the test piece, and watch for the antimony end point; it should be sharp but not always of the same intensity for different alloys. Twenty seconds after the end point is reached, remove the test piece, rinse with water, wipe to remove the deposit of antimony, and lightly scratch the surface with the sharp-pointed steel wire. If the coating has been removed, the surface will be readily scratched. Thirty seconds is sufficient for most unsealed coatings; sealed coatings will take from 30 sec. for coatings on the alloy 17S to 100 sec. for coatings on Alloy 52S. After the type of coating has been determined, proceed as described in the following Paragraph (*b*).

(*b*) Cut a small specimen from the sample to be tested (a specimen 1-in. square is convenient). Inscribe three circles, $\frac{5}{16}$ in. in diameter, number to identify, and measure the thickness of the test specimen at the center of the circles by means of a micrometer. Average the three readings. Any variations of reading greater than 0.0002 in. should be discarded and a new reading made. Adjust the level of the bath to its original volume by addition of water, bring to a boil and suspend the test specimen by means of a 2S aluminum wire. Care should be exercised that the wire does not touch any of the measured surfaces. The approximate time of stripping will have been determined in the preliminary test; 20-sec. immersion after the antimony deposit has formed should be sufficient to remove all coatings. Remove the test specimen, rinse quickly, wipe dry, care being used to remove the antimony from the surface. Measure the thickness of the test specimen at the center of the circles previously inscribed, average the readings, subtract from the first average, and divide by two (for specimens coated on both sides) to give thickness of coating.

Measurements of Coating Thickness:

A series of measurements of coating thickness as made in different laboratories by the different methods is included in Table I, in order to give an idea of the accuracy of these methods. The agreement between the coating thickness as determined by the stripping method and by measurement with a microscope, is remarkably close. However, the operator using the stripping method had had previous experience with it, so that his data are representative of about the best that can be done in this way. They are, moreover, sufficiently close for most practical purposes.

The stripping method gives directly what might be called the average coating thickness; however, if the coating varies appreciably in thickness from place to place, the thickness as determined by means of the microscope may well depend upon the number of readings taken and the care used to select representative places for measurement. As a rule, oxide coatings on aluminum are remarkably uniform in thickness; however, there may be an appreciable variation from point to point where the coated metal is an aluminum alloy, and this is particularly true in the case of aluminum-alloy castings. In Figs. 1 and 2 are shown sections of oxide coatings on commercial aluminum and on an alloy casting.

RESISTANCE OF OXIDE COATINGS TO ABRASION

The resistance of an oxide coating to abrasion, or its wearing quality, is frequently a matter of considerable practical importance. There are a number of factors contributing to abrasion resistance and their relation may not be a simple one. Some difficulty may be experienced, therefore, in correlating measurements made by different methods. Furthermore, the characteristics of oxide coatings are sufficiently different from those of paint, and varnish films that methods of measurement suitable for the latter may not be suitable for the measurement of the abrasion resistance of oxide coatings. While the oxide forming the coating is intrinsically very hard, the coating itself possesses a certain limited degree of friability which must be taken into account in measuring abrasion resistance and its use.

TABLE I.—MEASUREMENTS OF THICKNESS OF ANODIC COATINGS ON ALUMINUM.
Aluminum Research Laboratories

Sample[a]	Measurement by Microscope				Measurement by Calipers Before and After Stripping	
	Thickness, in.				Thickness, in. (Average of Two Sides)	
	Numbered Side		Unnumbered Side		First Set	Second Set
	Maximum	Average	Maximum	Average		
No. A-5	0.000168	0.000152	0.000168	0.000152	0.0002	0.0002
No. B-5	0.000312	0.000296	0.000312	0.000304	0.00035	0.00035
No. C-5	0.000448	0.000440	0.000448	0.000440	0.0005	0.0005
No. D-5	0.000608	0.000600	0.000608	0.000592	0.0006	0.0006
No. E-5	0.000176	0.000168	0.000176	0.000176	0.00025	0.0002
No. F-5	0.000328	0.000320	0.000336	0.000328	0.00035	0.00035
No. G-5	0.000480	0.000456	0.000488	0.000456	0.0005	0.0005
No. H-5	0.000616	0.000584	0.000640	0.000624	0.00065	0.0007

[a] Samples Nos. A, B, C and D are of Alloy 2S-H; samples Nos. E, F, G and H are of Alloy 53S-T.

Two typical methods of measurement have been compared in this investigation. Both of these methods measure the amount of abrasive effort required to wear through the coating. The one employs a rotating abrasive wheel and measures the revolutions required to abrade through the coating. The other method measures the number of grams of abrasive in an abrasive air blast required to penetrate the coating.

Abrasive Air Blast Method:

In this method an air blast containing a powdered abrasive is directed against the coating until it is penetrated. Schuh and Kern,[1] of Bell Telephone Laboratories, have studied the variables involved in such a method and have devised equipment and standardized the conditions necessary to make this a practical method for measuring the abrasion resistance of paint, varnish, and lacquer films. It also seems adapted to the measurement of the abrasion resistance of oxide coatings. According to their recommendations, carborundum powder of uniform particle size (170–200 mesh) is admitted at a constant rate (24 g. per min.) to a directed stream of air under a constant pressure of about 6 cm. of mercury, and the resulting carborundum-air blast is allowed to impinge upon the test surface mounted at an angle of 45 deg. and touching the end of the air blast nozzle. The weight of carborundum required to wear through the coating is taken as the abrasion resistance of the coating.

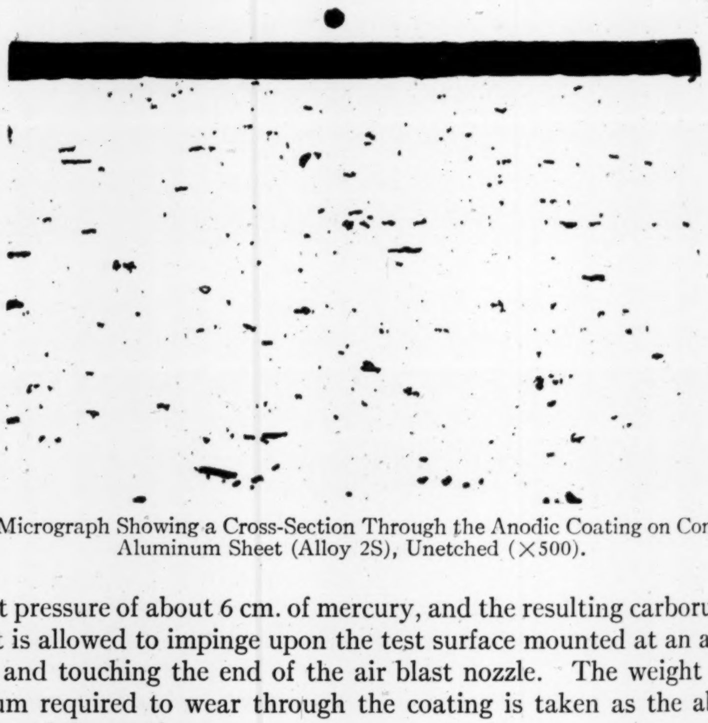

FIG. 1.—Micrograph Showing a Cross-Section Through the Anodic Coating on Commercial Aluminum Sheet (Alloy 2S), Unetched (×500).

Rotating Wheel Abrasiometer:

In this method, the number of revolutions of the test specimen against an abrasive wheel which are required to wear through a coating under standard operating conditions is taken as a measure of the abrasion resistance of the coating. In using this method, the conditions described by

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[1] A. E. Schuh and E. W. Kern, "Measurement of Abrasion Resistance," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 3, p. 72 (1931).

Camp[1] were employed. The surface to be tested must be in the form of a flat plate which can be mounted on a turn-table revolving at about 30 r.p.m. A small abrasive wheel (about 1.5 in. in diameter) is mounted at an angle of 60 deg. to the radius of the turn-table and rests on the coated surface under a constant and predetermined pressure. The abrasive wheel is freely mounted on a spindle, so that as the coated specimen revolves beneath and in contact with the abrasive wheel, the wheel also revolves and, being set at an angle, constantly abrades the surface in a path about $\frac{3}{8}$ in. in width as the specimen revolves. The number of revolutions of the specimen required before the abrasive wheel cuts through the oxide coating is taken as a measure of the abrasion resistance of the coating. It is, of course, essential that such factors as the speed of the specimen, the character of the wheel and the pressure on it, all be carefully standardized. The principal difficulty, however, in the use of this method arises from the difficulty of maintaining constant the abrasive action of the wheel. The importance of this factor is well illustrated by the experimental observations reported in Table II.

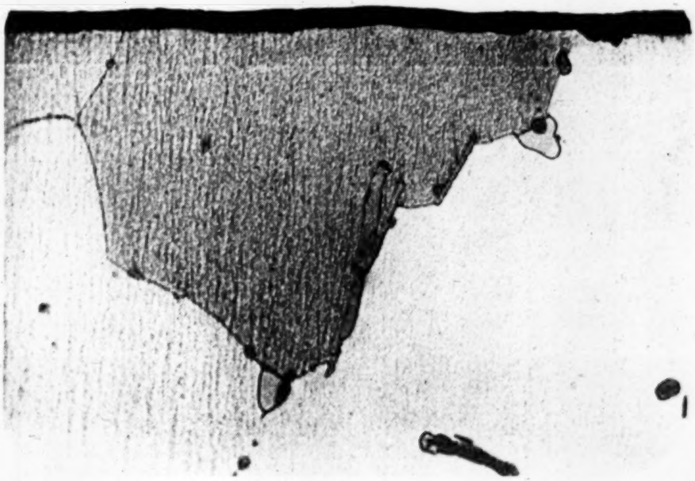

FIG. 2.—Micrograph Showing a Cross-Section Through the Anodic Coating on a Heat-Treated Aluminum Alloy Casting, Keller's Etch (×500).

Measurement of Resistance to Abrasion:

In Table II are reported series of measurements of the resistance to abrasion made with the rotating wheel abrasiometer method. The first series of measurements reported was made with an abrasive wheel fabricated with a rubber binder. The abrasive action of this wheel was sufficiently low so that substantial differences were observed in the abrasion resistance of different coatings, and from this standpoint the wheel was well adapted to differentiate between coatings of different resistance to abrasion. It was found, however, that such a wheel rapidly lost its abrading power when rubbed against these hard oxide coatings; this change was noticeable even in a few hundred revolutions. As a result, the same wheel would show greater resistance to abrasion against the reverse side of the specimen than it did against the side initially measured. These differences might be small in the case of the thinner or softer coatings, but might amount to as much as 100 per cent or more in the case of a thick coating. The ratio of abrasion resistance, as measured by the average number of revolutions of the specimen to the thickness of the coating, therefore varied from 14 to 318. As other measurements will show, this ratio should be more nearly constant, and the observed variation is largely due to the reduction in abrasive action of the wheel with continued operation rather than to any such variation in the hardness of the coating.

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[1] A. D. Camp, "Chipping and Abrasion Tests for Paint Coatings on Metal," *Industrial and Engineering Chemistry*, Vol. 20, p. 851 (1928).

TABLE II.—DETERMINATION OF RESISTANCE OF OXIDE COATINGS TO ABRASION BY ROTATING-WHEEL METHOD.

Sample	Abrasion Resistance by Rotating-Wheel Method					
	Revolutions of Alundum Wheel[a] Required to Wear Through the Coat		Ratio of Average Abrasion Resistance to Thickness[b] mils × 10²	Revolutions of Crystolon Wheel[a] Required to Wear Through the Coat		Ratio of Average Abrasion Resistance to Thickness[b] mils × 10²
No. A	260	240[c]	16	100	100[c]	6.6
No. B	300	530	14	140	150	4.8
No. C	1 500	1 250	32	180	190	4.2
No. D	1 600	1 925	30	250	250	4.2
No. E	770	880	48	75	80	4.5
No. F	2 390	3 550	92	210	240	6.9
No. G	4 830	7 450	135	350	310	7.2
No. H	10 270	28 060	318	430	470	7.5

[a] The alundum wheel was made with rubber binder to give low abrasive effect; the crystolon wheel was made with hard inorganic binder to give increased abrasive effect.

[b] The thickness values used in calculating this ratio are given in Table I under the column headed "Measurement by Microscope."

[c] The second value in this column is for a measurement made on the reverse side of the coating.

To minimize this loss in the cutting action of the wheel, a fused alumina wheel made with a hard inorganic binder instead of the rubber binder was substituted, and the wheel and the specimen were lubricated with kerosine during the operation of the test. With this hard wheel, the number of revolutions required to cut through the coating was substantially reduced and, although more difficulty was experienced in observing the end point, much more consistent results were secured in this way. The ratio of abrasion resistance to thickness of coating was more nearly constant with the different coatings and in this respect resembled the measurements made by the abrasive air-blast method.

While it might be possible, by selecting a hard abrasive wheel, to make the rotating wheel method suitable for the measurement of the abrasion resistance of oxide coatings, the mechanical difficulties of maintaining the wheel true and with a satisfactory cutting face are sufficiently real to warrant against its use when the more readily reproducible abrasive air-blast method is available.

The resistance of the same series of oxide coatings to abrasion was measured by the abrasive air-blast method in two different laboratories; the results are given in Table III. The apparatus used differed in mechanical details, and there were differences in the operating conditions, as indicated by the footnotes to Table III. Nevertheless, the results from the two laboratories are in fairly close numerical agreement. Because of substantial differences in the experimental conditions, the question may well be raised as to whether this agreement is not accidental. The work of Schuh and Kern[1] was confined to the measurement of the resistance of

TABLE III.—RESISTANCE OF OXIDE COATINGS TO ABRASION AS DETERMINED BY THE ABRASIVE AIR-BLAST METHOD.

Sample	Tests by Bell Telephone Laboratories					Sample	Tests by Aluminum Company of America				
	Grams of Carborundum Required to Wear Through Coat[a]				Ratio of Abrasion Resistance to Thickness, in mils × 10²		Grams of Alundum Required to Wear Through Coat[b]				Ratio of Abrasion Resistance to Thickness, in mils × 10²
No. A-1	23	24	26	25		No. A-6	28	35	32	28	
No. A-2	27	30	26	28			30	28	28	30	
No. A-3	31	28	30	28		Average	29.9				1.97
Average	27.1				1.78						
No. B-1	66	65	66	68		No. B-6	71	74	71	73	
No. B-2	68	69	72	70			72	72	71	73	
No. B-3	73	75	74	73		Average	72.1				2.40
Average	69.9				2.33						
No. C-1	94	98	97	99		No. C-6	115	111	106	110	
No. C-2	97	102	97	100			110	111	108	110	
No. C-3	106	105	104	103		Average	110.2				2.50
Average	100.2				2.28						
No. D-1	123	125	129	126		No. D-6	149	150	148	147	
No. D-2	126	129	130	126			147	146	148	148	
No. D-3	133	136	137	139		Average	147.9				2.48
Average	129.9				2.18						
No. E-1	44	42	45	45		No. E-6	36	35	36	35	
No. E-2	40	39	40	41			35	34	34	35	
No. E-3	41	39	40	41		Average	35.0				2.03
Average	41.4				2.41						
No. F-1	76	77	80	80		No. F-6	77	80	80	76	
No. F-2	80	84	89	86			80	79	81	78	
No. F-3	86	87	92	85		Average	78.9				2.44
Average	83.5				2.58						
No. G-1	103	102	109	104		No. G-6	125	123	116	116	
No. G-2	111	111	114	114			119	120	120	119	
No. G-3	115	116	114	113		Average	119.8				2.63
Average	110.5				2.42						
No. H-1	143	143	147	148		No. H-6	161	163	160	160	
No. H-2	154	153	155	165			165	160	162	160	
No. H-3	158	161	157	160		Average	161.4				2.67
Average	153.7				2.54						

[a] The method, as operated by the Bell Telephone Laboratories, employs 180-mesh carborundum in an air blast under a pressure of 10 cm. of mercury. The specimen is set in contact with the end of the air-blast nozzle at an angle of 45 deg. through the vertical nozzle. The first two abrasion values in each horizontal column were measured on the front of the specimen, the second two values on the reverse side.

[b] The method, as operated by the Aluminum Company of America, employs 180-mesh alundum in an air blast under a pressure of 5.17 cm. of mercury (approximately 1 lb. per sq. in.). The nozzle is set $\frac{1}{4}$ in. from the surface of the specimen and with the specimen at an angle of 45 deg. to the vertical nozzle. The four values in the first horizontal column for each specimen were measured on the front face of the specimen, the second column of four on the reverse side.

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[1] *Loc. cit.*

lacquer, varnish, and enamel films to abrasion and the relative importance of the various test conditions may be substantially different when the test is employed on oxide coatings than when it is used to measure the resistance of softer paint and varnish films. For example, Schuh and Kern found that increasing the air pressure from 5 cm. to 10 cm. of mercury cut in half the weight of abrasive required to wear through a lacquer coating. They also found that raising the nozzle from the specimen increased somewhat the weight of abrasive required to penetrate a lacquer coating.

In the tests just described, the higher air pressure employed by the Bell Telephone Laboratories might be expected to lower substantially the weight of abrasive required, and this should more than compensate for the effect of raising the nozzle from the specimen, as was done in the equipment employed in the laboratory of the Aluminum Company of America. In the one series of tests, carborundum was employed as the abrasive and in the other alundum. The numerical coincidence, therefore, of the results from the two laboratories may not be significant. However, the ratio of abrasion resistance to thickness, as indicated in Table III, shows good correspondence by the two methods. The values for the two thinnest coatings are slightly lower than the ratios for the thicker coatings. They are close enough, in any case, to suggest that the resistance to abrasion is substantially proportional to the thickness of the coating, and increases somewhat as the total coating thickness increases.

ELECTRICAL BREAKDOWN VOLTAGE

One of the potential uses for oxide coatings is in the insulation of electrical conductors. In certain applications, the chemical resistance and incombustible character of the oxide coating combined with dielectric strength are very desirable. An A.S.T.M. method of test for the determination of dielectric strength[1] is available for the measurement of the insulating value of oxide coatings. The method, as applied to an oxide-coated sheet of aluminum, simply involves making an electrical connection to the sheet at one edge, after scratching through the oxide coating, and then placing an electrode in contact with the coating, applying an e.m.f. and increasing it at a uniform rate until the oxide coating breaks down and is punctured by the electrical discharge.

It is customary to use a flat-faced electrode of substantial area in testing insulating varnish films. As might have been anticipated, the breakdown voltages of these oxide coatings measured in this way were found to depend in an important degree on the area contacted by the electrode. The largest electrode gave the lowest breakdown voltage (see Table IV).

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[1] This test is described in Sections 11 to 15 of the Tentative Methods of Testing Varnishes Used for Electrical Insulation (A.S.T.M. Designation: D 115 - 36 T) of the American Society for Testing Materials, see *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1039 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 1028.

The oxide coatings apparently have points of weakness, at which breakdown occurs, and these points may be of relatively small area and of substantially lower breakdown voltage than most of the coating. If they are few in number, the chance of hitting one with the point contact of the ball-shaped electrode is small, but increases as the area of contact of the electrode is increased. For most practical purposes the minimum breakdown voltage is of primary interest and this can be readily ascertained by employing a flat electrode of appropriate contact area. In this connection the relation between the size of the test specimen and the area of the flat electrode is an important factor. An electrode should be selected with which it is possible conveniently to explore most of the surface of the test specimen and at the same time make a sufficient number of measurements to obtain a true estimate of the general character of the oxide film. In the current study 4 by 4-in. specimens were used. From the viewpoint considered and in the light of the data obtained, it appears that an electrode of from ½ to 1 sq. in. is quite satisfactory for test specimens of this size.

TABLE IV.—DIELECTRIC BREAKDOWN VALUES IN VOLTS, 60-CYCLE ALTERNATING CURRENT.

Sample	General Electric Co.	Bell Telephone Laboratories	Aluminum Research Laboratories[a]	
	Ball-Shaped Electrode, Point Contact	Flat Electrode, Contact Area of 1 sq. in.	Flat Electrode, Contact Area of 2.5 sq. in.	Ball-Shaped Electrode, Point Contact
No. A	283	234	220	220
No. B	567	290	238	385
No. C	643	363	220	422
No. D	1053	483	256	662
No. E	228	224	220	238
No. F	368	258	220	294
No. G	435	321	256	366
No. H	655	392	330	495

[a] In making these measurements, the potential was not increased continuously after the initial application but step-wise in steps of 110 v. These data, as well as the other values in the table, are the averages of a number of individual observations.

The breakdown voltage may also be used as a method of measuring the film thickness of the coating. For this purpose a spherical-tipped electrode providing point contact with the surface of the coating might be employed. In this case the data indicate that, excluding the breakdown values obtained from the 2.5 sq. in. flat electrode, the breakdown voltage varies directly with the film thickness.

In addition to giving a good approximation of film thickness, a breakdown test might be quite adequate for exposing erratic behavior of the film resulting from porosity, non-uniformities in film thickness, processing conditions and the nature of the alloy. For this purpose a flat electrode should be employed. It is interesting to note in this connection that even the thickest films contain weak spots as revealed by the data obtained with the 2.5 sq. in. electrode. The photomicrographs, Figs. 1 and 2, illustrate the fact that the breakdown voltage might vary from point to point for reasons outlined above.

CONCLUSION

The foregoing report, of course, deals only with a part of the methods available for measuring the properties of thickness, abrasion resistance, and electrical breakdown voltage of oxide coatings on aluminum and, of course, further experience with the methods presented will be necessary before standardized conditions of operation can be established and the accuracy of the methods adequately appraised. Furthermore, it is recognized that other methods, particularly for control purposes, are demanded by industry and will have to be developed to meet these requirements. This report, therefore, should be considered as a progress report in this rapidly developing field.

Respectfully submitted on behalf of Subcommittee VI,

JUNIUS D. EDWARDS,
Chairman.

REPORT OF COMMITTEE C-1
ON
CEMENT

Although Committee C-1 on Cement held but one meeting during the past year, much has been accomplished and the work now in progress indicates that there will be much data available at the 1938 meeting of the Society, which may result in further recommendations to the Society. The interest of the public in all types of hydraulic cements, and especially in more critical test methods and requirements, has been reflected in the committee's activities.

Recommendations Affecting Standards:

As a result of the year's work, the committee is submitting one new specification, a revision of two tentative standards, revisions for immediate adoption in two standards, and the adoption as standard of a portion of an existing tentative standard as outlined in subsequent sections of the report. The standards covered by these recommendations are listed in the table showing the results of the letter ballot vote, given at the close of the report.

High-Early-Strength Cements:

The Sponsoring Committee on High-Early-Strength Cement (M. A. Swayze, chairman) has nothing of particular interest to present at this time. At the last meeting of the Society the committee recommended the adoption as standard of the Specifications for High-Early-Strength Portland Cement (C 74 – 36) after certain revisions. The revision of most moment was that concerning the definition. In the standard as it now appears additions, other than calcium sulfate and/or water, not exceeding 1 per cent are permissible provided they are shown not to be harmful through tests prescribed and carried out by Committee C-1. In the section of this report dealing with the work of the Cement Reference Laboratory, more regarding this phase of the work of Committee C-1 will be given, but it is desirable here to call attention to the fact that the committee is now testing one addition to cements which have been produced at three mills.

Masonry Cements:

The committee is not yet ready to make any changes in the status of the Tentative Specifications for Masonry Cement (C 91 – 32 T). It is recommending that these specifications be retained as tentative for another year. During the past year a revision of the Federal specification for this commodity has become effective. The Federal standard contains several requirements not included in the Society's specifications and demands considerably higher strengths. As a result, much discussion and study have arisen and it is rather desirable that before any changes be made in the Society's standard the reactions to the requirements of the Federal standard should be followed.

The extended use of cements containing predominating amounts of slag for the setting of masonry has led the sponsoring committee to gather data for use in preparing a specification for this type of cement. The first draft of such a proposed standard is now being critically studied.

The Sponsoring Committee on Masonry Cement (H. D. Baylor, chairman) has revised one of the early standards of the Society, namely, the Standard Specifications for Natural Cement (C 10 – 09). The committee is recommending the withdrawal of this old standard and the immediate adoption of the new revised specifications appended hereto,[1] and accordingly asks for the necessary nine-tenths vote at the annual meeting in order that this recommendation may be referred to Society letter ballot.

Portland Cements:

The committee is recommending the deletion of Section 3 on fineness from the Standard Specifications for Portland Cement (C 9 – 30).[2] This paragraph covers the use of the No. 200 sieve for the determination of the fineness of portland cement. Cement made according to present processes has such a slight residue on this sieve that the residues no longer have any significance as a measure of true fineness. The Sponsoring Committee on Portland Cement (W. H. Klein, chairman) is giving thought to both the need of any requirements for fineness in cement specifications and to observing the use and results of the turbidimetric measurements specified in the Standard Specifications for High-Early-Strength Portland Cement (C 74 – 36). This Sponsoring Committee has no other recommendations to make at this time. It is following very closely the investigations being carried out by the working committees studying volume changes, particularly those changes resulting from steam treatment at high pressures and temperatures, and the use of plastic mortars. The deletion of Section 3 in Standard Specifications C 9, mentioned above, is recommended for immediate adoption and the committee accordingly asks for the necessary nine-tenths vote at the annual meeting in order that this recommendation may be referred to letter ballot of the Society.

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[1] These revised specifications were adopted as standard by the Society and appear in the 1937 Supplement to Book of A.S.T.M. Standards, p. 52.—ED.

[2] This revision was adopted by the Society and the revised specifications appear in the 1937 Supplement to book of A.S.T.M. Standards, p. 49.

Blended Cements:

The Working Committee on Blended Cements (R. E. Roscoe, chairman) has fulfilled its promise of having ready for presentation to the Society proposed Tentative Specifications for Blended Cements, which are appended hereto.[1] The increasing marked interest in such cements has made it rather imperative that the committee have the standard available at an early date, and it is gratifying to note the progress made. The wide variety of materials used in this cement has not lessened the troubles of carrying out this work. The attempt to cover such a wide variety in one standard and to base an acceptance strength requirement on a concrete test will no doubt lead to considerable discussion of these tentative specifications. Such discussion will be welcome, but it is hoped that those who take part will have in mind the problems which confronted the committee and in offering adverse criticism on any portion of the standard will be sure to offer something constructive.

Fineness:

It will have been noted above that the committee is recommending the deletion of the requirement for the use of the No. 200 sieve for the fineness test prescribed in the Standard Specifications for Portland Cement (C 9 - 30). The reasons for this action are given in the report by the Working Committee on Fineness (J. C. Pearson, chairman) appended hereto.

It can be clearly seen from the report of the Working Committee and the general testing procedure that the tendency at the present time is to express fineness in so-called specific surface. There are faults in such a procedure and the committee is well aware of them. But with the decrease in the average size of the particle which has been brought about by modern grinding methods and which has rendered meaningless the results obtained with the No. 200 sieve (and for many cements the No. 325 sieve) some new method was needed and expressing fineness as the area per unit weight had considerable to commend it.

In the present transitional stage, the Society's standards for hydraulic cements exhibit quite a difference in fineness requirements. The specifications for natural cements (C 10) still requires the No. 200 sieve; those for high-early-strength cement (C 74) as well as the newly proposed tentative specifications for blended cements require that fineness be determined by the use of the turbidimeter; while the specifications for portland cement (C 9) after revision this year will contain no requirement covering fineness. These facts illustrate the need for the active work which the Working Committee on Fineness is pursuing. In the meantime, the committee recommends the continuance as tentative for another year of the Tentative Method of Test for Fineness of Portland Cement by Means of the Turbidimeter (C 115 - 34 T).

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[1] The proposed tentative specifications, see page 291, were not accepted by the Society at the annual meeting. They are being given further consideration in the committee.—ED.

Plastic Mortars:

The committee is recommending the adoption of the revisions proposed by the Working Committee on Plastic Mortars (O. L. Moore, chairman) in the Tentative Method of Tests for Compressive Strength of Portland Cement Mortar (C 109 – 34 T). These revisions cover Sections 3, 4(*b*), 6, 10, 13, and 14 of the methods. The reasons for the proposed changes are given in the appended report of the Working Committee. The committee further recommends the retention of the revised methods, appended hereto,[1] as tentative for another year.

The Working Committee will devote its activities during the coming year to trying out a concrete instead of a mortar as an acceptance test for cement. This is referred to briefly in the report of the Working Committee. What work has been done so far clearly indicates that this concrete is far more plastic than the mortar defined in the Tentative Methods C 109 – 34 T. In spite of its containing aggregate passing a $\frac{3}{8}$ in. and retained on a No. 4 sieve, it can be readily molded into a 2-in. cube and yields a specimen but little subject to water losses through bleeding during the setting of the cement. While many may feel that a 1:5 by weight "gap graded" concrete is far from an average concrete, yet it is decidedly nearer to such than a 1:3 mortar. The use of the concrete as an acceptance test for cement has much to commend it and the securing of standard materials for such a purpose offers no really great difficulties.

Volume Changes:

The Working Committee on Volume Change and Soundness of Portland Cement (H. F. Gonnerman, chairman) has been actively continuing its studies of volume changes induced by subjecting bars of plastic paste after the first 24 hr. hardening under normal conditions to a period of treatment at elevated steam temperatures and pressures. The need for further intensive studies of the effect of such treatment is shown by the recent announcement of the majority of the cement manufacturers of the country through the Portland Cement Association, that very shortly the members of the Association will ship no cement which has been found to have an expansion in excess of 1 per cent when subjected to a high-temperature – pressure treatment developed by the Association. An outline of the test is appended to this report.[2]

The chairman of the Working Committee has reported that since the last report the Working Committee has been making further studies of the autoclave test, particularly with a shorter steaming cycle. The data presented in the report of last year[3] were based on total steaming cycles of 24, 48, and 72 hr. and a maximum temperature of 350 F. (175 C.).

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[1] See p. 726.
[2] See p. 290.
[3] *Proceedings*, Am. Soc. Testing Mats. Vol., 36, Part I, p. 225 (1936).

The desirability of having a shorter period for an accelerated test led to the investigation of a shorter cycle which has been used during the current year. This cycle comprises a total period of 5 hr. in the autoclave and includes a 1-hr. rise to the maximum temperature of 420 F. (215 C.) and a 1-hr. cooling period. Recent studies have included a comparison of this cycle with the longer periods at 350 F. (175 C.) described in the former report. Some tests were also made using a 3-hr. cycle holding the specimens 1 hr. at the maximum temperature of 420 F. (215 C.).

Attention has been given to the development of suitable equipment for carrying out autoclave tests in accordance with a definitely prescribed procedure. Since such equipment is now available commercially, the Working Committee is inaugurating a series of cooperative tests in ten laboratories which has for its main objective the development of a suitable standardized procedure for making such tests. Among the variables to be studied in this cooperative series are the maximum temperature and duration of the steaming cycle, length of specimens, age at test, consistency of the mix, time required for cooling the specimens before measuring for length, and the uniformity of results obtained in different laboratories on the same cements and on specimens tested on different days.

Chemical Analysis:

The Working Committee on Methods of Chemical Analysis (W. C. Hanna, chairman) has been actively pursuing its work of trying out new methods in cooperation with cement chemists throughout the country. Its work has advanced to such a degree that it recommends the adoption as standard of Sections 1 to 10 of the Tentative Method of Chemical Analysis of Portland Cement (C 114 – 35 T), after some revisions, thereby also revising Sections 8, 9, 10 and 11 of the Standard Methods of Sampling and Testing Portland Cement (C 77 – 32),[1] mostly editorial in nature. The quick methods of determining magnesia as given in Sections 11 to 14 of Methods C 114 – 35 T will be retained as tentative for another year. Procedures for the determination of manganese and phosphorus, as appended hereto,[2] are being recommended for publication as tentative for inclusion in Methods C 114.

The Working Committee has inaugurated a comprehensive cooperative study of methods of determining the oxides of sodium and potassium in cement, and has given thought to developing methods for determining sulfides, free lime, and ferrous iron. A report of the Working Committee, including the new methods for determining manganese and phosphorus, is appended hereto. Copies of the methods being used for the determination of the alkali oxides can be secured by addressing the chairman of this Working Committee.

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[1] This recommendation was adopted by the Society and the revised methods appear in the 1937 Supplement to Book of A.S.T.M. Standards, p. 54.

[2] See p. 717.

Cement Reference Laboratory:

The report of the Cement Reference Laboratory accompanying this report tersely shows the activity of this laboratory. Particular attention is called to the increased number of laboratories desiring inspection in the present fifth tour and to the inauguration of the work which Committe C-1 has given the laboratory of determining whether an addition other than calcium sulfate and/or water to high-early-strength portland cement is harmful. The laboratory also distributed a sample of cement to a large number of laboratories which had expressed a desire to engage in the cooperative tests. The results are presented in a report prepared by the Cement Reference Laboratory.[1] Pending a collation of comments from the laboratories which engaged in that test, none are offered herewith, excepting that it is quite evident that better agreement among laboratories was obtained than in possibly any other previous cooperative tests.

Tests of Additions to Cement:

It was necessary to create the new Subcommittee on Tests of Additions to Cement as a result of the adoption of the revised Standard Specifications for High-Early-Strength Portland Cement (C 74 – 36). The duties of this subcommittee were to determine the tests needed to find out whether an addition was harmful, in a general way to outline the test procedure and after completion of the tests to analyze the data, and finally decide whether the additions under investigation were not harmful. This subcommittee, under Inge Lyse, chairman, held an organization meeting at the laboratory of the Pennsylvania State Highway Department on August 31, and September 1, 1936. Present at part or all of the meetings were: Messrs. F. H. Jackson, H. S. Mattimore, C. H. Scholer, and Inge Lyse of the subcommittee, Mr. P. H. Bates, chairman of C-1 Committee, Messrs. C. L. Warwick, G. E. Warren, Cloyd M. Chapman, G. L. Lindsay, C. T. Raber and J. R. Dwyer. Mr. Dwyer acted as secretary of the subcommittee. A proposed general program of tests for additions to cement was prepared. The details of the test methods and procedures were left to a considerable extent in the hands of the Cement Reference Laboratory. The items of the test program included:

1. Sampling of the cements, with and without additions,
2. Fineness of the cements,
3. Percentage of addition,
4. Standard cement tests,
5. Storage tests,
6. Age of concrete,
7. Types of test specimens, cylinders and mortar beams,
8. Concrete mixes,
9. Air voids determination,

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[1] See Report on Cooperative Tests of Portland Cement Comparative Sample, C.R.L. No. 1, published in ASTM BULLETIN, December, 1937.

10. Consistency of concrete,
11. Freezing and thawing of mortars,
12. Sulfate resistance,
13. Volume changes,
14. Bleeding, and
15. Water solubility.

The Cement Reference Laboratory is making tests of one type of addition to three brands of high-early-strength portland cement and it has received inquiries regarding such tests from several other concerns.

Recommendations Affecting Standards:

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 71 members; the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. PROPOSED TENTATIVE STANDARD			
Specifications for Blended Cements[a]	42	17	8
II. PROPOSED REVISIONS OF TENTATIVE STANDARDS			
Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 – 34 T)	59	2	5
Method of Chemical Analysis of Portland Cement (C 114 – 35 T), new procedures for determining manganese and phosphorus in cement	63	0	5
III. PROPOSED REVISIONS OF STANDARDS			
Specifications for Portland Cement (C 9 – 30), immediate adoption[b]	57	8	3
Specifications for Natural Cement (C 10 – 09), immediate adoption	52	5	11
IV. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Method of Chemical Analysis of Portland Cement (C 114 – 35 T), Sections 1 to 10 as modified editorially	63	0	5

[a] The classified vote on the Proposed Tentative Specifications for Blended Cement was as follows: Affirmative: 17 producers, 15 consumers, 10 general interests; negative: 10 producers, 4 consumers, 3 general interests. The recommendation for publication as tentative of these specifications failed of approval at the annual meeting of the Society.

[b] The classified vote on the adoption of the proposed revision of the Standard Specifications for Portland Cement (C 9 – 30) was as follows: Affirmative: 27 producers, 19 consumers, 11 general interests; negative: 2 producers, 2 consumers, 4 general interests.

This report has been submitted to letter ballot of the committee which consists of 71 members; 62 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

P. H. BATES,
Chairman.

L. W. WALTER,
Secretary.

REPORT OF WORKING COMMITTEE ON FINENESS

At the meeting of Committee C-1 on July 1, 1936, a proposal to include in the cement specification a wet method for determining the residue of cement on the No. 200 sieve was introduced by the Port of New York Authority, represented by Mr. Sinnickson. This proposal was referred to the Working Committee on Fineness, which in the meantime has given careful consideration to the method. While the committee recognizes the advantages of the wet method in the saving of time and labor as compared with the dry method, it wishes to emphasize the point to which attention has been called by practically all investigators in this field; namely, that all portland cements are now so finely ground that sieve residues are meaningless, except for certain control operations in the manufacturing process. Therefore, the Working Committee recommends that the time-honored requirement for a fineness test on the No. 200 sieve, by either wet or dry methods, be now abandoned.

This recommendation for the withdrawal of the fineness test of cement on the No. 200 sieve is based on the information and data given in the following Paragraphs 1 to 5:

1. Data from fineness tests of cement that have been presented to the committee on numerous occasions, of which the following tabulation is a typical example:

Specific Surface, sq. cm. per g.	Range in No. 200 Sieve Residue, per cent
1440 to 1480	5.3 to 14.2
1700 to 1750	1.9 to 7.4
2050 to 2250	0.1 to 6.8
2480 to 2610	1.1 to 6.1

No. 200 Sieve Residue, per cent	Range in Specific Surface, sq. cm. per g.
0.1 to 2.0	1700 to 2610
2.2 to 3.5	1730 to 2260
4.6 to 5.4	1440 to 2480
6.1 to 7.0	1700 to 2520
7.4 to 7.7	1470 to 1730
10.6 to 14.2	1450 to 1480

The table shows that sieve residues in the finest group as indicated by specific surface overlap those in the coarsest group, and that a very wide range of sieve residues correspond to any given value of specific surface, and conversely. In other words, the sieve residues bear little or no relation to the fineness as indicated by specific surface.

2. Closed-circuit grinding, now common in the industry, lowers very materially the No. 200 sieve residue as compared with open-circuit grinding, for any given value of specific surface.

3. It is assumed that the committee desires to recognize the technical advance that has been made in measuring the fineness of normal and high-early-strength cements, and that it will accordingly be willing to discard hopelessly outmoded and ineffective test methods.

4. Organizations other than this Society are abandoning the No. 200 sieve test in their cement specifications.

5. The general opinion is that the present fineness requirement in the cement specifications has been meaningless for many years. This requirement should either be eliminated, or if a fineness test is deemed necessary by a majority, it should be replaced by some method that furnishes dependable information on particle size distribution, specific surface, or minimum percentage of some fine fraction designated by maximum particle size.

Respectfully submitted on behalf of the working committee,

J. C. PEARSON,
Chairman.

REPORT OF WORKING COMMITTEE ON PLASTIC MORTAR TESTS

The Working Committee on Plastic Mortar Tests is giving consideration to a proposed concrete compressive strength method and is submitting herewith revisions in the Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (A.S.T.M. Designation: C 109-34 T).

Proposed Concrete Test:

A description of the proposed concrete test was presented in June, 1936, to the members of the Working Committee. After considerable correspondence and experimentation an outline of tests has been agreed upon and sixteen laboratories are now participating in a cooperative series of tests. The investigation covers two mixes, each having one part by weight of cement to about five parts of other solids. One of these mixes was proposed by P. H. Bates and the other by J. C. Pearson. Gravel of low absorption (0.4 per cent) graded from No. 4 to $\frac{3}{8}$ in. in size and standard Ottawa sand graded between sieves Nos. 20 to 30 are used in specified quantities in both mixes. In addition, the Bates mix will contain a specified weight of potter's flint while the Pearson mix will contain a given weight of Ottawa banding sand. A cement-water ratio by weight of 1.88 ($W/C =$ 0.8 by volume) is specified. A given consistency as determined by the one-half size slump cone will be required and will be obtained by changing, if necessary, the cement paste content. The regular mix specified in Methods C 109 will be included for comparative purposes. Flow tests using the 10-in. flow table and a Burmister flow trough, as modified by Lewis H. Tuthill, will be made in order to study the suitability of these apparatus for measuring consistency. Three samples of portland cement, a standard cement, a high-early-strength cement and a moderate-heat cement will be used. The test specimens will be 2-in. cubes and the test periods 1, 3, 7, and 28 days, tests to be repeated on three days. The cement, low absorption pebbles, potter's flint, and banding sand will be prepared and distributed to the participating laboratories which will furnish the standard Ottawa sand.

Recommended Revisions of Mortar Test Method:

In December, 1935, a copy of the proposed revisions in Sections 3, 4 (*b*), 10, 11, 13 and 14 of the Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 - 34 T)[1] was presented to

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 743 (1934); also 1936 Book of A.S.T.M. Tentative Standards, p. 393.

Committee C-1 and discussed. The proposed revision of Section 3 on molds was based primarily upon studies made by the Cement Reference Laboratory, which studies were not finished at that time but have since been published in a paper entitled "Effect of Departure from Planeness of Bearing Surfaces on the Compressive Strength of 2-in. Mortar Cubes," by J. R. Dwyer.[1] These completed studies support the requirements of the proposed revision.

The change in Section 4 (*b*) on testing machine involves a revision of the footnote and was suggested by H. F. Gonnerman to improve the accuracy of centering 2-in. cubes when using a $3\frac{1}{8}$-in. upper bearing block convenient in testing larger specimens. In Section 4 (*b*) it is stated that the diagonal or diameter of the spherical upper bearing block shall be only slightly greater than the diagonal of a 2-in. cube while in the footnote it is stated that $3\frac{1}{8}$ in. is satisfactory, etc. Experience shows that this diameter for the upper bearing block is too large for accurate centering of 2-in. cubes unless the lower bearing block is smaller. Since machine manufacturers are supplying lower filler bearing blocks in several diameters to suit the size of the specimen, the proposed change in the footnote is an improvement and brings the method in line with practice. The description of the tests for sieve analysis of graded Ottawa sand in Section 7 has been considerably improved.

The proposed revisions of Sections 10, 11, 13 and 14 are based upon rather complete studies presented to the committee in December, 1935, report. The principal revision is that of Section 11 introducing more definite methods of molding specimens. The proposed changes in Sections 10, 13 and 14 are principally changes in wording to clarify the meaning of the sections. More recently, the Federal Specifications Executive Committee has included a compression test in its Federal Methods for Sampling, Inspection, and Testing of Hydraulic Cements (SS-C-158) and has adopted methods of test similar to those in the Tentative Standard C 109.

In addition to the above changes a new Section 1 on Scope has been added to the methods which has changed the section numbering. All of the changes discussed above have been incorporated in the completely revised methods appended hereto[2] and the Working Committee by unanimous vote recommends that the methods as revised be continued as tentative for another year.

Respectfully submitted on behalf of the Working Committee,

O. L. MOORE,
Chairman.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 351 (1936).
[2] See p. 726.

REPORT OF WORKING COMMITTEE ON METHODS OF CHEMICAL ANALYSIS OF PORTLAND CEMENT

In previous reports there have been discussed the criticisms which had been received of the Tentative Method of Chemical Analysis of Portland Cement (C 114 – 35 T).[1] However, no further suggestions have been received since the 1936 report.[2] Since there seems to be general satisfaction with the method, the Working Committee recommends that Sections 1 to 10, inclusive, of Method C 114 – 35 T be adopted as standard with certain minor revisions of an editorial nature and incorporated in the Standard Methods of Sampling and Testing Portland Cement (C 77 – 32)[3] to replace Sections 8, 9, 10 and 11 of Methods C 77.[4]

It is recommended that Sections 11 to 14, inclusive, of Method C 114 – 35 T, which describe two methods for the rapid determination of magnesia, be continued as tentative for another year. No criticism of these two quick methods has been received, but it is believed that they should be retained as tentative methods a while longer. It is hoped that the members of Committee C-1 will study them and let the Working Committee have the benefit of any criticism that they may wish to make.

Study of Tests for Manganese and Phosphorus in Cement:

It was not possible to include in the 1936 report the results of the investigation of methods for manganese and phosphorus because a few of the cooperating laboratories had not finished their work. The studies have now been completed and the results are listed in Table I. The names of the cooperating laboratories are as follows:

Idaho Portland Cement Co., W. R. Chandler, Chief Chemist,
National Bureau of Standards, Division V-4, G. E. F. Lundell, Chief,
Tennessee Valley Authority, P. J. Freeman, Principal Materials Engineer,
California Portland Cement Co., O. D. Guire, Jr., Analyst,
Portland Cement Assn., H. F. Gonnerman, Manager of Laboratory,
Universal Atlas Cement Co., Duluth Plant, T. A. Hicks, General Chemist,
Universal Atlas Cement Co., Buffington Plant, T. A. Hicks, General Chemist,
Riverside Cement Co., T. F. Mullan, Chief Chemist, and
Monolith Portland Cement Co., O. C. Hart, Chief Chemist.

NOTE.—The order of laboratories does not correspond to the tabulation of results.

With a single exception the results for Mn_2O_3 are remarkably good. Those for P_2O_5 while not quite as consistent are satisfactory as compared with results received in previous investigations. As a whole the analysts approved of the methods, but a few of them made suggestions for improvement. Before the investigation was started, G. E. F. Lundell of the National Bureau of Standards assisted the Working Committee with a critical study of the methods and suggested several changes. His staff also carried out some of the analytical work.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 766 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 382.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 222 (1936).

[3] 1936 Book of A.S.T.M. Standards, Part II, p. 9.

[4] This recommendation was adopted by the Society and the revised methods appear in the 1937 Supplement to Book of A.S.T.M. Standards, p. 54.

Another method for manganese was suggested by H. F. Gonnerman of the Portland Cement Association during the course of the investigation.[1] This method as modified was tested in the laboratories of the Universal Atlas Cement Co., and the California Portland Cement Co. It was found to be less satisfactory than the method now recommended as tentative[2] because of difficulty in recognizing the endpoint of the titration in the presence of a large precipitate.

TABLE I.—ANALYTICAL DATA REPORTED BY COOPERATING LABORATORIES ON DETERMINATIONS OF MANGANESE AND PHOSPHORUS IN CEMENT.

Samples *G*, *H*, and *J* are portland cements for general use. The brands are well known and were selected in order to have a sample high in Mn_2O_3, one high in P_2O_5, and one approximately of the average composition of American cements.

Laboratory	Mn_2O_3[a]			P_2O_5[b] Not Corrected for Blank			P_2O_5 Corrected for Blank		
	Sample G	Sample H	Sample J	Sample G	Sample H	Sample J	Sample G	Sample H	Sample J
1	0.11	0.85	0.06	0.17	0.08	0.69			
2	0.11	0.87	0.07	0.16	0.08	0.72	0.12	0.04	0.68
3	0.10	0.86	0.06	0.14	0.03	0.68	0.11	nil	0.65
4	0.10	0.85	0.07	0.07	0.04	0.65	0.05	0.02	0.63
5	0.11	0.99	0.07	0.13	0.01	0.66			
6	0.10	0.87	0.06	0.11	0.01	0.61	0.10	nil	0.60
7	0.11	0.85	0.07	0.14	0.04	0.64			
8	0.10	0.85	0.06	0.11	0.04	0.69	0.07	nil	0.65
9	0.12	0.89	0.08	0.11	0.02	0.65			
Average	0.11	0.88	0.07	0.13	0.04	0.67	0.09	0.01	0.64

[a] Method for Mn_2O_3: The manganese in the solution of cement is oxidized to permanganic acid with sodium bismuthate and then titrated with sodium arsenite.

[b] Method for P_2O_5: The phosphorus in the solution of cement is precipitated as ammonium phosphomolybdate and then reprecipitated as magnesium ammonium phosphate.

Cooperating through C. H. Scholer, Professor of Applied Mechanics, W. A. Van Winkle, Department of Chemistry, Kansas State College of Agriculture and Applied Science, is not only making alkali determinations for the Working Committee, but also has made an independent study of the problem of the determination of manganese and phosphorus. For the manganese determination he used a sample of cement of his own and the samples of cement G, H and J furnished by the Working Committee. After testing the method recommended by the committee for approval as tentative and comparing it with a colorimetric method he reported consistent and checking results.

Van Winkle also made determinations of phosphorus in samples G, H and J and a fourth sample of cement. He tried the method proposed as tentative[1] and a volumetric method. Although he obtained good results with the proposed method, he experienced some difficulty due to the precipitation of silica and ammonium molybdate with ammonium phosphomolybdate. He thought it was due to the high temperature and great acidity prevailing at the time of adding ammonium molybdate to precipitate phosphorus as phosphomolybdate. He thought that the volumetric method on the other hand would give excellent results and require less time.

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[1] Ford's modification of method described on page 1612 of Scott's "Standard Methods of Chemical Analysis," Fourth Ed.

[2] See p. 722.

As a result of the data which have been obtained the committee recommends that the Methods for Determining Phosphorus and Manganese in Cement, as appended hereto,[1] be accepted for publication as tentative and included in the Tentative Method C 114.

Study of Test for Alkali in Portland Cement:

In accordance with instructions received last June, a cooperative investigation of methods for the determination of alkali in portland cement is now in preparation. This is the fourth series of cooperative studies to be undertaken by the Working Committee. Its preparation has required much thought and correspondence. A critical examination of some of the methods has been made by G. E. F. Lundell of the National Bureau of Standards, and P. S. Roller of the U. S. Bureau of Mines has improved the method which he and A. A. Berk had published.

The laboratory work has only recently been started and the program will involve considerable work. The Working Committee is indebted to the following who are cooperating in this investigation:

1. Portland Cement Assn., H. F. Gonnerman, Manager of Laboratory,
2. Tennessee Valley Authority, P. J. Freeman, Principal Materials Engineer,
3. Universal Atlas Cement Co., T. A. Hicks, General Chemist,
4. Edison Cement Corp., G. C. Wilsnack, Chief Chemist,
5. Coplay Cement Manufacturing Co., W. J. Johnson, Chief Chemist,
6. Superior Cement Corp. (Ohio), L. I. Loghry, Chief Chemist,
7. California Division of Highways, T. E. Stanton, Materials and Research Engineer,
8. Kansas State College of Agriculture and Applied Science, C. H. Scholer, Professor of Applied Mechanics,
9. The Olympic Portland Cement Co., Ltd., F. H. Ronk, Chief Chemist,
10. California Portland Cement Co., O. D. Guire, Jr., Analyst,
11. Idaho Portland Cement Co., W. R. Chandler, Chief Chemist,
12. Giant Portland Cement Co., S. G. McAnally, Chief Chemist, and
13. Yosemite Portland Cement Co., H. McC. Larmour, Chief Chemist.

Copies of the four methods being studied can be obtained from the chairman.

Chairman P. H. Bates suggested that the Working Committee consider R. B. Rudy's method for the determination of sulfuric anhydride in portland cement by means of the Wagner turbidimeter, published by the National

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[1] See p. 722.

Bureau of Standards.[1] A member of this committee, T. A. Hicks, had one of his chemists, L. G. Sprague of the Universal Atlas Cement Co., Northampton, Pa., make two series of tests. The published procedure was followed in all details in the first series but it seemed that the precipitation of $BaSO_4$ was not complete during the stirring period prescribed by the method. In the second series the precipitate was stirred at a slightly higher speed for 60 sec. instead of 30 sec. The results of Mr. Sprague's series of tests were as follows:

	Sample	SO_3 by Turbidimeter	SO_3 by Gravimetric Method	Error
First Series	1	1.90	1.87	+0.03
	2	1.82	1.86	−0.04
	3	1.86	1.91	−0.05
	4	1.72	1.75	−0.03
	5	1.82	1.81	+0.01
	6	1.84	1.82	+0.02
	7	1.92	1.99	−0.07
Second Series	1	1.90	1.87	+0.03
	2	1.85	1.86	−0.01
	3	1.87	1.91	−0.04
	4	1.78	1.75	+0.03
	5	1.79	1.81	−0.02
	6	1.83	1.82	+0.01
	7	1.97	1.99	−0.02

Chairman P. H. Bates has called attention to a procedure for determining silica in cement which is also described in a Bureau of Standards Research Paper.[2] Mr. T. A. Hicks had this method tested at two plant laboratories of the Universal Atlas Cement Co. Although the method was reported to give good results, it is the opinion of the Working Committee that if a rapid method is desired for the determination of silica, the well known and commonly used perchloric acid method is preferable until more study can be made.

In conclusion, the Working Committee desires to indicate its gratitude to those who participated in the investigation of methods for manganese and phosphorus, to G. E. F. Lundell for his constructive criticisms, and also to the Universal Atlas Cement Co. and California Portland Cement Co. for making it possible for members of their staffs to continue to assist in this work.

Respectfully submitted on behalf of the Working Committee,

W. C. HANNA,
Chairman.

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[1] Robert B. Rudy, "Determination of Sulfuric Anhydride in Portland Cement by Means of the Wagner Turbidimeter," National Bureau of Standards *Journal of Research*, Vol. 16, No. 6, June, 1936, p. 555.

[2] Edwin E. Maczkowske, "A Rapid Method for the Determination of Silica in Portland Cement," National Bureau of Standards *Journal of Research*, Vol. 16, No. 6, June, 1936, p. 549.

REPORT OF SUBCOMMITTEE ON CEMENT REFERENCE LABORATORY

(Period July 1, 1936, to March 31, 1937)

Requests were received by the Cement Reference Laboratory for inspection of 34 cement testing laboratories. The total number of laboratories now involved in the present inspection tour is 296, a larger number than included in any of the preceding tours. The following tabulation affords a comparison of the numbers and classifications of the laboratories involved in the various tours:

Classification of Laboratories	Number of Laboratories Inspected in Tour No. 1	No. 2	No. 3	No. 4	No. 5
Cement producers	99	108	104	100	120
Commercial testing laboratories	45	48	30	37	66
State highway laboratories[a]	25	65	53	61	63
Schools	21	27	20	13	23
Municipal	11	10	9	6	10
Miscellaneous	7	7	8	12	14
Total	208	265	224	229	296

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[a] Includes highway branch laboratories, some of which are located at cement mills.

Inspections were made at 107 laboratories. Since the commencement of the present tour in May, 1936, there have been performed 141 inspections; about one-half of the field inspections thus far listed for the current tour have accordingly been completed.

The territory already covered includes the northeastern section of the country and many laboratories in Ohio and in the vicinity of Chicago.

Some few requests have been recently received for inspections to be made of laboratories located in the territory which has just been visited. It is not contemplated that any early revisits will be made to such localities. Some of these requests were from laboratories which are probably interested in work which is under the supervision of government offices which insist on Reference Laboratory inspection. It is probable that more requests will be received from commercial testing laboratories.

The U. S. Bureau of Public Roads and the Federal Emergency Administration of Public Works have both expressed considerable interest in the cement laboratory inspections and copies of 56 of the Reference Laboratory inspection reports have been furnished to them. The issuance of such copies, as in the past, is conditional upon proper authorization from the inspected laboratories. An illustration of the extent to which a government office may use these reports is afforded by a recent instance in which approval as a testing agency was withheld from a certain laboratory until a more satisfactory type of moist storage equipment was installed.

Reports were received from nearly all of the laboratories which received portions of the comparative sample, distributed by the Reference Laboratory some months ago. These results were received during the midst of field inspection work and were only completely summarized at a recent date.[1] Copies of the tabulation of results are being distributed, through the Bureau, to the cooperating laboratories and also to the members of Committee C-1. Consideration was given as to how the data might be most appropriately presented. It is realized that, if such comparative tests are found advisable as a regular procedure, the results must be reported to the laboratories soon after completion of the tests. Therefore, comments are being invited from the laboratories and from the members of Committee C-1 so that, if such tests are to be repeated, the summaries of results may quickly be prepared and reported.

This subcommittee met with the Subcommittee on Tests of Additions to Cements and prepared a recommended procedure to be followed in arranging for the tests of cements containing additions made subsequent to calcination. These tests are required by the recently adopted Standard Specification for High-Early-Strength Portland Cement (C 74 – 36). The Society has approved the method for authorizing and reporting such tests, and arrangements were recently made by an applicant to have such a series of tests made on cements containing a certain addition. The Reference Laboratory is securing the samples and the tests are under way.

In so far as the field work, sampling and other activities of the Reference Laboratory have permitted, some time was devoted to the investigation of the stirring apparatus for the Wagner turbidimeter. As a result of trials and observations on various types of stirrers, one has just recently been designed which has given a promising performance and will be studied further as soon as possible.

Information is being obtained regarding compression testing machines observed during the laboratory inspections. These brief details are not only interesting, but they should prove helpful in planning any calibration work of such machines, provided developments make it necessary for the Reference Laboratory to participate in such calibration. This question will receive further consideration during the next few months.

Respectfully submitted on behalf of the subcommittee,

G. E. WARREN,
Chairman.

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[1] See Report on Cooperative Tests of Portland Cement Comparative Sample, C. R. L. No. 1, published in ASTM BULLETIN, December, 1937.

APPENDIX I

PROCEDURE FOR MAKING AUTOCLAVE TESTS OF PORTLAND CEMENT USED AS BASIS OF COMPARISON IN CURRENT COOPERATIVE STUDIES

STUDIED BY WORKING COMMITTEE ON VOLUME CHANGE AND SOUNDNESS

Preparation of Test Bars.[1]—Mix a neat cement paste of normal consistency in accordance with the methods described in Sections 19 to 22 of the Standard Methods of Sampling and Testing Portland Cement (A.S.T.M. Designation: C 77 - 32).[2] Immediately after mixing, fill the mold in three layers. Compact each layer with the rubber glove-protected forefinger, working from one end of the mold to the opposite end in 25 strokes (12 strokes for 5-in. bar). Work along the bar and back two times or a total of four times for each layer. After the top layer has been compacted smooth the specimen with a few strokes of the trowel leaving the paste slightly above the sides of the molds.

Immediately after the mold has been filled, place it in the moist closet or moist room at a temperature of 70 F. ± 3 F. (21 C. ± 1.6 C.) and a relative humidity of at least 90 per cent. Approximately 2 hr. after molding give the bars the final troweled finish, mark the identifying reference numbers on the surface and continue the moist curing. At 22 to 23 hr. after molding remove the bars from the molds and place in water at 80 F. (27 C.).[3] Approximately 1 hr. after removal from the molds, surface dry the bars, one at a time, with a cloth and immediately weigh[4] and measure for length in a length comparator. After weighing and measuring, keep the bars in water until placed in the autoclave.

Autoclaving.—At the age of 24 hr. place the bars in the autoclave and heat the water rapidly to bring the temperature of the steam in 1 hr. to 420 F. (215 C.) which corresponds to a gage pressure of 295 lb. per sq. in. Care should be taken to permit the air to escape from the autoclave during the heating to 212 F. (100 C.). Maintain the 420 F. (215 C.) temperature for 3 hr. After the desired period in the autoclave at the 420 F. (215 C.) temperature, shut off the heat supply and allow the autoclave to cool slowly so that the pressure will gradually fall to near atmospheric by the end of 1 hr. At the end of the 1-hr. period release slowly the small pressure remaining through a vent valve until atmospheric pressure is attained. Then open the autoclave and allow the bars to cool for 15 min. in the autoclave; and then cool them for 15 min. in the air of the laboratory. After cooling the bars in air, immerse them in water which has been preheated to the temperature of the bars (approximately 150 F. (66 C.)). Gradually cool the water and bars to 80 F. (27 C.)[3] in a 30-min. period and then surface dry, weigh, and measure the bars for length. Express the change in length during autoclaving, as a percentage of the length before placing in the autoclave.

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[1] The test bars recommended for these cooperative studies are 1 by 1 by 11¼ in. long with stainless steel inserts and an effective gage length of 10 in.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 9.

[3] If desired, a water temperature of 70 F. (21 C.) instead of 80 F. (27 C.) may be used.

[4] Weighing recommended for future reference.

APPENDIX II

PROPOSED
TENTATIVE SPECIFICATIONS AND METHODS OF TEST
FOR
BLENDED CEMENTS[1]

Scope

1. These specifications cover hydraulic cements known as "blended cements" in which portland cement is an essential constituent. It does not include cements intended for masonry purposes only.

SPECIFICATIONS

Definition

2. A blended cement is the finely divided product obtained by intimately mixing together, preferably by intergrinding, portland cement and one or more admixtures (other than the material used to regulate the set of portland cement) which are added to alter one or more properties of the cement.

Admixtures

3. The admixtures may consist of the following:

(*a*) Pozzolanic materials, either natural or artificial,

(*b*) Cementitious materials, such as limes, natural cements and other calcium silicate cements,

(*c*) Accelerators, plasticizers, retarders and waterproofing materials,

(*d*) Inert non-deleterious siliceous materials.

Quality of the Ingredients

4. (*a*) Any material used in compounding a blended cement shall conform to the definition or specification of the American Society for Testing Materials, where such definition or specification has been adopted by the Society.

(*b*) *Pozzolanic Materials.*—A substance is said to be pozzolanic when, while not necessarily cementitious by itself, it possesses constituents which will combine with calcium hydrate at ordinary temperatures in the presence of moisture to form stable compounds of cementitious value.

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[1] These proposed tentative specifications were submitted by Committee C-1 on Cement to the Society at the annual meeting but were not accepted. They are being given further consideration in the committee.—ED.

(*c*) *Accelerators, Plasticizers, Etc.*—The producer must guarantee that any accelerator, plasticizer, retarder, waterproofing agent, or any other materials not otherwise covered by these specifications, be free from any deleterious ingredient which might adversely affect the quality of the concrete.

(*d*) *Inert Admixture.*—When an inert admixture is used, it must not retard or hasten the hardening of portland cement. It must be insoluble in water and in a solution of calcium hydroxide. It shall not contain anything that may be injurious to the durability of the concrete.

Producer's Statement

5. When requested by the purchaser, the manufacturer shall state in writing the nature, origin and amount of each of the materials used, furnish satisfactory proof as to their quality and describe the process followed in compounding the product to be furnished under these specifications.

Fineness

6. (*a*) The specific surface shall not be less than 1800 sq. cm. per gram of cement.

(*b*) The residue on a standard No. 325 sieve shall not exceed 15 per cent by weight.

Soundness

7. (*a*) A pat of neat cement of normal consistency shall remain firm and hard and show no signs of cracking, checking or disintegration when subjected to the steam test for soundness as described in Standard Methods C 77.[1]

(*b*) A pat of neat cement of normal consistency, molded in Le Chatelier tongs, after 24-hr. storage in a standard moist closet shall not show more than 5-mm. expansion when subjected to the steam test for soundness as described in Standard Methods C 77.[1]

(*c*) The average expansion of three 1 by 1 by 10-in. bars of neat cement of normal consistency shall not be greater than 0.20 per cent when subjected to the autoclave test described in Section 18.

(*d*) Three 1 by 1 by 10-in. bars of standard plastic mortar shall remain firm and hard and show no signs of distortion, cracking, checking or disintegration when subjected to the steam test for soundness as described in Standard Methods C 77.[1] Further, they shall not show as a result of the steaming an average expansion or contraction greater than 0.05 per cent.

Sulfate Resistance

8. If the purchaser so desires he may require that the cement be tested for sulfate resistance, in which case, in addition to the tests for soundness prescribed in Section 7, the average increase in the length of 3 additional bars, each 1 by 1 by 10-in. in length and made of 1 part cement

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[1] 1937 Supplement to Book of A.S.T.M. Standards, p. 54.

and 5 parts standard Ottawa sand, shall not exceed 0.075 per cent after storage for 1 day in the moist closet, 2 days in water, and 25 days in a solution containing 5 per cent sodium sulfate (Na_2SO_4) and 5 per cent magnesium sulfate ($MgSO_4$).

Time of Setting

9. The cement when tested in standard plastic mortar shall not develop initial set in less than 60 min. nor final set in more than 10 hr. as determined by the Vicat needle in accordance with Section 21.

Compressive Strength

10. (*a*) The average compressive strength of not less than three 3 by 6-in. concrete cylinders shall be not less than 1000 lb. per sq. in. at 7 days and 2000 lb. per sq. in. at 28 days.

(*b*) In case the purchaser desires a cement developing high-early-strength, he may specify that the concrete develop an average compressive strength of 1000 lb. per sq. in. at 1 day and 2000 lb. per sq. in. at 3 days. The purchaser may also require that the compressive strength of the concrete shall be not less than 3000 lb. per sq. in. at 28 days.

(*c*) In case the purchaser specifies a cement having low heat of hydration (Section 11) the average compressive strength of concrete shall be not less than 750 lb. per sq. in. at 7 days and 1500 lb. per sq. in. at 28 days.

(*d*) The compressive strengths obtained at 28 days shall be greater than those obtained at 7 days, and in the case of the high-early-strength cement the strengths at 3 days shall be greater than the strengths at 1 day.

Heat of Hydration

11. (*a*) The heat of hydration shall not exceed 75 cal. per g. at 7 days and 85 cal. per g. at 28 days when tested in accordance with the method described in Sections 23 and 24.

NOTE.—Emphasis is placed on the fact that the method for determining the heat of hydration as described in Section 23 is intended for portland cement. Some of the blended cements might contain material which is dissolved with difficulty, in which case complete solution may not be obtained in this test.

(*b*) The purchaser, if he desires, may specify that the heat of hydration shall not exceed 65 cal. per g. at 7 days and 75 cal. per g. at 28 days. In this event, however, the average compressive strength of the concrete may be lower but shall be not less than 750 lb. per sq. in. at 7 days and 1500 lb. per sq. in. at 28 days as provided in Section 10 (*c*).

Packaging and Marking

12. (*a*) When, as specified, the cement is delivered in packages, the words "Blended Cement" and, in addition, the nature of the blend, the name of the manufacturer, the brand, and the weight of the cement contained therein shall be plainly marked on each package. When, as specified,

the cement is delivered in bulk shipments, this information shall be contained in the shipping advices accompanying the shipment.

(*b*) A bag shall contain 94 lb. net. A barrel shall contain 376 lb. net. All packages shall be in good condition at the time of inspection.

Storage

13. The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment, and in a suitable weather-tight building which will protect the cement from dampness.

Inspection

14. Every facility shall be provided the purchaser for careful sampling and inspection at either the mill or at the site of the work, as may be specified by the purchaser. At least 6 days from the time of sampling shall be allowed for completion of the 1-day tests; at least 8 days shall be allowed for completion of the 3-day tests; at least 12 days shall be allowed for completion of the 7-day tests; and at least 33 days shall be allowed for completion of the 28-day tests. The cement shall be tested in accordance with the methods hereinafter prescribed.

Rejection

15. (*a*) The cement may be rejected if it fails to meet any of the requirements of these specifications.

(*b*) Cement remaining in storage prior to shipment for a period greater than 6 months after completion of the tests shall be retested and shall be rejected if it fails to meet any of the requirements of these specifications.

(*c*) Cement failing to meet any of the tests for soundness specified in Section 7 may be accepted if it passes a retest using a new sample at any time within 28 days thereafter.

(*d*) Packages varying more than 5 per cent from the specified weight may be rejected; and if the average weight of packages in any shipment, as shown by weighing 50 packages taken at random, is less than that specified, the entire shipment may be rejected.

METHODS OF TESTING

Methods of Sampling and Testing

16. The cement shall be sampled and tested in accordance with the Standard Methods of Sampling and Testing Portland Cement (A.S.T.M. Designation: C 77) of the American Society for Testing Materials,[1] unless otherwise specified or provided for in these specifications.

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[1] 1937 Supplement to Book of A.S.T.M. Standards, p. 54.

Fineness Test

17. The specific surface shall be determined in accordance with the Tentative Method of Test for Fineness of Portland Cement by Means of the Turbidimeter (A.S.T.M. Designation: C 115 – 34 T) of the American Society for Testing Materials.[1]

Autoclave Test

18. (*a*) A neat cement paste of normal consistency shall be mixed in accordance with the methods described in Sections 25 to 28 of the Standard Methods of Sampling and Testing Portlant Cement (A.S.T.M. Designation: C 77) of the American Society for Testing Materials.[2] Immediately after mixing, the 1 by 1 by 10-in. mold shall be filled with the paste in three layers, each layer being compacted with the rubber-glove-protected forefinger, working from one end of the mold to the opposite end in 25 strokes, along the bar and back twice or a total of four times for each layer. After the top layer has been compacted, the specimen shall be smoothed off with a few strokes of the trowel, leaving the paste slightly above the sides of the mold. Immediately after the mold has been filled it shall be placed in the moist closet. Approximately 2 hr. after molding, the specimens shall be removed from the moist closet and given the final troweled finish, identifying numbers being inscribed on the surface by means of dies. The specimens in the molds shall then be replaced in the moist closet.

(*b*) Twenty-two hours after molding the specimens shall be removed from the molds and placed in water at 80 F. (27 C.). Approximately 1 hr. after removal from the molds, the specimens, one at a time, shall be surface-dried with a cloth and immediately measured for length in a dial gage length comparator which is graduated to 0.0001 in. After being measured, the specimens shall be kept in water until placed in the autoclave at the age of 24 hr.

(*c*) While in the autoclave the bars shall be held in a rack so that each is exposed to saturated steam in the same manner. Steam shall be generated from a charge of water, about 1 in. in depth, below the specimens in the autoclave. The water shall be heated rapidly to bring the temperature of the steam in 1 hr. to 420 F. (215 C.), which corresponds to a gage pressure of 295 lb. per sq. in. This temperature shall be maintained constant, by means of a regulator, for 3 hr. After this 3-hr. period the heat supply shall be shut off and the autoclave allowed to cool, the pressure being gradually reduced to atmospheric by the end of 1 hr. The cylinder shall then be opened and the specimens allowed to cool for 15 min. in the autoclave and another 15 min. in air to about 150 F. (66 C.). The specimens shall then be immersed in water which has been preheated to 150 F. (66 C.)

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 777 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 399.

[2] 1937 Supplement to Book of A.S.T.M. Standards, p. 54.

or to the temperature of the bars. The water shall then be gradually cooled during a 30-min. period to 80 F. (27 C.). The bars, one at a time, shall then be removed from the water, surface dried and quickly measured.

Soundness Test of Standard Plastic Mortar

19. The mortar shall be proportioned, mixed, and molded in accordance with the Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (A.S.T.M. Designation: C 109 – 37 T) of the American Society for Testing Materials,[1] except that when molding, instead of tamping, each layer shall be puddled with the finger tips protected by snug-fitting rubber gloves. The specimens shall be stored and steamed in accordance with the Standard Methods of Sampling and Testing Portland Cement (A.S.T.M. Designation: C 77) of the American Society for Testing Materials,[2] the specimens being removed from the molds and measured just prior to the steam test.

Sulfate Resistance Test

20. (*a*) The percentage of water used to fabricate the 1:5 mortar bars described in Section 8 shall be calculated by using the following formula:

$$\text{Percentage of water (in terms of dry weight of sand and cement)} = \frac{2P}{3(N+1)} + 6.5.$$

where P = the normal consistency of the cement used, and
N = the parts of standard Ottawa sand. The quantity of water shall be expressed in milliliters.

(*b*) The volume of the salt solution prescribed in Section 8 shall not be less than 2 liters for each three bars immersed in it.

Time of Setting Test

21. (*a*) A standard Vicat mold (see Section 23 of Standard Methods C 77),[2] resting on a glass plate, shall be filled with the standard plastic mortar and stored in a moist closet. This mortar shall be proportioned, mixed and molded as prescribed in Section 19.

(*b*) By initial set is meant that point in the hardening of the mortar when it begins to harden and thereafter progressively continues to lose its plasticity or semifluid condition.

(*c*) Initial set shall be said to have occurred when a Vicat needle 1 mm. in diameter and weighing 300 g. fails to penetrate to the bottom of a Vicat ring filled with standard plastic mortar and thereafter at 15-min. intervals continues to progressively penetrate to a less and less depth.

(*d*) By final set is meant that point in the hardening of the mortar when the same Vicat needle fails to penetrate the surface of the plastic mortar specimen.

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[1] See p. 726.

[2] 1937 Supplement to Book of A.S.T.M. Standards, p. 54.

Compressive Strength

22. The concrete for the compression test specimens shall be mixed with a water-cement ratio of 1.0 and it shall have a slump of from 2 to 4 in. The coarse aggregate shall be sound and well graded. The sand shall be graded. The cement-sand relation by volume should be held close to 1:2. The procedure for mixing, molding, and testing of the specimens shall conform to that described in the Standard Methods of Making Compression Tests of Concrete (A.S.T.M. Designation: C 39) of the American Society for Testing Materials.[1]

Heat-of-Hydration Test[2,3]

23. (*a*) *Apparatus.*—The apparatus shall include a 1-pt. open-mouth thermos bottle with cork stopper, a Beckmann thermometer having a range of about 6 C., a magnifying meniscus reader for the thermometer, glass funnel, glass rod stirrer, analytical balance for weighing the sample, and a balance accurate to 0.2 g. for weighing the calorimeter and solvent. A glass stirring rod shall be bent to give an arm at the lower end about 1 in. in length and shall be flattened at an angle of 45 deg. to form a propeller. The stirrer shall extend almost to the bottom of the thermos bottle and shall be operated at a speed of about 400 r.p.m.

The stirrer, the immersed section of the Beckmann thermometer and the inside of the thermos bottle shall be coated with a suitable material, such as a resin diluted with an equal quantity of acetone, to protect the glass against the hydrofluoric acid. The thermometer shall be so adjusted that its upper range approximates room temperature.

(*b*) *Solvent.*—A mixture of nitric and hydrofluoric acids shall be used to dissolve the cement and the hydrated pastes. A large batch of about 2.0 *N* HNO_3 shall be prepared, using 127 ml. of 70 per cent HNO_3 (sp. gr. 1.42) per liter. It is not necessary to standardize this HNO_3 solution, but the heat capacity of the system shall be determined each time a new batch of HNO_3 solution is prepared.

The hydrofluoric acid shall be added when the calorimeter sample is prepared, using 8 ml. of 48 per cent HF for each determination.

(*c*) *Preparation and Curing of Specimen.*—A 300-g. sample of cement and 120 g. of distilled water shall be mixed together and vigorously stirred with a mechanical stirrer for 5 min., after which a representative portion shall be used to fill four[4] or more homeopathic vial containers ($\frac{3}{4}$ by 4 in.) in approximately equal amounts. The temperature of the materials shall be regulated, commensurate with mixing-room temperature, so that the temperature of the cement paste shall be 70 F. ± 5 F. (21 C. ± 2.75 C.). The vials shall be sealed immediately upon filling with a tightly fitting cork which in turn shall be coated with paraffin. The vials shall be stored in a vertical position at 70 F. ± 1 F. (21 C. ± 0.55 C.) until the samples are tested. At each of the ages of 7 and 28 days, the heat of solution of one specimen of the partially hydrated cement in a given acid mixture shall be determined.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 342.

[2] This method for determining the heat of hydration is intended for portland cement.

[3] For a discussion of this heat-of-hydration test, see paper by William Lerch, "A Simple Apparatus for Determining Heat of Hydration of Portland Cement," *Paper No. 29*, Portland Cement Association Fellowship at the National Bureau of Standards.

[4] This number provides for two companion specimens, over and above normal requirements, to allow for rechecking, breakage, etc.

Test Procedure

24. (*a*) *Heat Capacity of System.*—A quantity of the HNO_3 solution shall be cooled to the temperature corresponding to the lower range of the Beckmann thermometer and some of the solution placed in the thermos bottle. Eight milliliters of HF and then HNO_3 shall be added to give the desired weight, using 400 to 450 g. of the solvent. The cork stopper, through which pass the stirrer, funnel, and thermometer, shall be properly seated. The lower end of the funnel shall extend as much as $\frac{1}{8}$ in. below the lower surface of the stopper and shall be as much as $\frac{1}{2}$ in. above the top surface of the liquid. The stirrer shall have sufficient freedom of motion to prevent contact with the stopper.

The motor shall be started and the stirring continued for about 5 min. to attain uniform temperature distribution. The initial temperature, estimated to the nearest 0.001 C., shall then be recorded and 7 g. of ignited (700 to 800 C. for 1 hr.) zinc oxide shall immediately be introduced through the funnel, completing the addition in from 1 to 2 min. The temperatures shall be recorded at 5-min. intervals until the temperature change between successive intervals is constant. The time required for complete solution of the sample may vary from 10 to 20 min.

If small temperature changes occur after complete solution of the sample, due to stirring or heat leakage, a correction for these changes shall be applied to the temperature rise. Thus, if the time for complete solution is 20 min. and the temperature rises 0.005 C. in each 5-min. interval thereafter, 0.020 C. is deducted from the temperature rise attained in 20 min.

Using the value 257 cal. per g. as the heat of solution of ignited ZnO in the solvent, the heat capacity of the system shall be calculated as follows: The heat of solution of 7 g. of ZnO divided by the corrected temperature rise gives the heat capacity of the system in calories per degree Centigrade.

At the completion of the test, the calorimeter shall be opened and the stem of the funnel and the cork stopper observed. If more than a trace of the sample adheres to them, the test shall be rejected.

(*b*) *Heat of Solution of Cement.*—The determination of the heat of solution of the cement or the hydrated cement shall be made by the same procedure as described in Paragraph (*a*) for determining the heat capacity of the calorimeter, except that there shall be used a 3-g. sample of cement or an equivalent quantity of hydrated cement on the ignited weight basis. The time required for complete solution will vary with different cements and is about 10 to 25 min.

The temperature rise obtained on dissolving the cement shall be corrected for the heat of stirring as already described.

Since the partly hydrated cement contains a considerable quantity of water, an additional correction shall be made for the heat required to change the temperature of this water from room temperature to the final temperature of the calorimeter. Thus, if the room temperature is 25 C., the final temperature of the calorimeter 26 C., and the partly hydrated cement sample (which was introduced to the calorimeter at room temperature) contains 1 g. of water, then 1 cal. will have been required to raise the temperature of this water from room temperature to the final temperature of the calorimeter. Hence, 1 cal. shall be added to the previously calculated heat of solutions of the hydrated cement sample.

The heat of solution of the 3-g. cement sample shall be calculated by multiplying the corrected temperature rise by the heat capacity of the calorimeter, converting this result to the heat of solution per gram of cement by dividing by the quantity of dry, unignited cement in the calorimeter sample.

The quantity of dry, unignited cement contained in the damp calorimeter sample shall be calculated from ignition tests on corresponding dry and partly hydrated samples. This quantity shall be used as a divisor in place of the actual weight of the calorimeter sample for calculating the heat of solution of the partly hydrated cement from the total heat of solution determined in the test.

The heat of hydration of the cement shall be calculated by subtracting the heat of solution of the partly hydrated cement from the heat of solution of the original cement.

REPORT OF COMMITTEE C-3

ON

BRICK

Committee C-3 on Brick has held three meetings during the past year, in Atlantic City, N. J., on July 2, 1936; in Washington, D. C., on November 16, 1936; and in New York City on March 22, 1937.

The committee has been very active during the year. Several of its specifications have been revised and indications are that the specifications and methods of test under its jurisdiction are being used to a greater extent by consumers. A new set of by-laws has been adopted which it is believed will increase the operating efficiency of the committee. The subcommittees have been completely reorganized, the former subcommittees having been discharged and the following new subcommittees formed:

I. Advisory (P. H. Bates, chairman)
II. Clay Building Brick (D. E. Parsons, chairman)
III. Concrete Brick (W. H. Kaiser, chairman)
IV. Sand-Lime Brick (C. H. Carmichael, chairman)
V. Paving Brick (F. H. Jackson, chairman)
VI. Sewer Brick (T. R. Lawson, chairman)
VII. Glazed Building Units (H. E. Stringer, chairman)

Several new members have been added to the committee during the year. The present membership of the committee is 38, of whom 15 are classified as producer, 7 as consumer, and 16 as general interest members.

At the request of the Executive Committee of the Society, Committee C-3 considered the proposal to merge with Committee C-10 on Hollow Masonry Building Units.[1] At present, the fields of jurisdiction of the two committees are not clearly defined; also there are certain products which are of interest to both committees, such as glazed building units and double brick, and under one committee the question of jurisdiction over such materials would disappear. This proposal was referred to letter ballot of the committee which consists of 38 members; 26 members returned their ballots, of whom 18 voted affirmatively, 4 negatively, and 4 marked their ballots "not voting."

Subsequent to the 1936 annual meeting, Committee C-3 presented to the Society on August 26, 1936, through Committee E-10 on Standards proposed Tentative Specifications for Glazed Building Units (C 126 – 36 T),[2] proposed revisions of the Tentative Specifications for Building Brick (Made from Clay or Shale) (C 62 – 35 T) and of the Tentative Methods of Testing

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[1] See Editorial Note, p. 304.

[2] Subsequent to the 1937 annual meeting a revision of these specifications was presented to the Society through Committee E-10 on Standards, see Editorial Note, p. 304.—Ed.

Brick (Modulus of Rupture, Compressive Strength, Absorption) (C 67 – 35 T). These recommendations were accepted[1] and the new and revised tentative standards appear in the 1936 *Proceedings*.[2]

RECOMMENDATIONS AFFECTING STANDARDS

I. Proposed Tentative Standards.—The committee recommends that the proposed Tentative Specifications for Paving Brick, appended hereto,[3] be accepted for publication as tentative to replace the Standard Specifications for Paving Brick (C 7 – 30)[4] which in part no longer represent commercial practice. Accordingly, the Standard Specifications C 7 – 30 are recommended to be withdrawn. The new tentative specifications are based upon research conducted by the U. S. Bureau of Public Roads, Ohio State Highway Testing Laboratory and the National Paving Brick Association.

The committee also recommends that the proposed Tentative Specifications for Sewer Brick (Made from Clay or Shale), appended hereto,[5] be accepted for publication as tentative to replace the Standard Specifications for Clay Sewer Brick (C 32 – 24)[6] which in the light of information recently developed are obsolete. Accordingly, the Standard Specifications C 32 – 24 are recommended to be withdrawn. The new tentative specifications are based in part upon tests made at the National Bureau of Standards showing the relation of abrasive resistance to strength and water absorption of bricks and in part upon the results of a circularization of 15 sanitary engineers requesting their views on sewer brick specifications. Replies were received from 7 of the 15 engineers addressed.

II. Proposed Revision of Tentative Standard.—The committee recommends that the Tentative Specifications for Building Brick (Made from Clay or Shale) (C 62 – 36 T)[7] be completely revised as appended hereto[8] and continued as tentative. These tentative specifications as revised, as well as the specifications as revised in 1935 and 1936, are based upon data presented as part of the annual report of Committee C-3 in 1935.[9] The 1935 and 1936 editions of the specifications attempted to retain the classification used in the present Standard Specifications for Building Brick (Made from Clay or Shale) (C 62 – 30) and were criticized on account of the resulting complicated method of classification and grading. In the tentative specifications as revised this year, classifications based upon differences in strength have been abandoned completely.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee C-3 reported the following results of the letter ballot vote of a total of 26 ballots returned from a committee membership of 38: Specifications C 126, affirmative 21, negative 0, ballots marked "not voting" 5; Specifications C 62, affirmative 23, negative 0, ballots marked "not voting" 3; Methods C 67, affirmative 26, negative 0.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 776, 773, 780 (1936); also 1936 Book of A.S.T.M. Tentative Standards, pp. 428, 425, 432.

[3] See p. 740. Subsequent to the annual meeting, additional revisions of these specifications were presented to the Society through Committee E-10 on Standards, see Editorial Note on p. 304.—ED.

[4] 1936 Book of A.S.T.M. Standards, Part II, p. 125.

[5] See p. 749.

[6] 1936 Book of A.S.T.M. Standards, Part II, p. 134.

[7] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 773 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 425.

[8] See p. 733.

[9] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 243 (1935).

III. Revision of Standards.—The committee recommends for immediate adoption a revision of the Standard Specifications for Concrete Building Brick (C 55 – 34)[1] for the purpose of bringing the specifications into conformity with the testing procedure prescribed in the Tentative Methods of Testing Brick (C 67 – 36 T), and now being recommended for adoption as standard. The committee accordingly asks for the necessary nine-tenths vote at the annual meeting in order that this revision, given below, may be referred to letter ballot of the Society for immediate adoption.

Section 4.—Change the fourth and fifth sentences to read as follows by the addition of the italicized words and the omission of those in brackets:

> For the purpose of tests, not less than [ten] *five* bricks shall be required for each investigation. In general, two samples of [ten] *five* bricks each shall be tested for every 100,000 bricks contained in the lot under consideration; but where the total quantity exceeds 500,000 bricks, one sample of [ten] *five* bricks from each 100,000 bricks shall be tested.

The committee also recommends for immediate adoption a revision of the Standard Specifications for Sand-Lime Building Brick (C 73 – 30),[2] likewise for the purpose of bringing the specifications into conformity with the testing procedure prescribed in Methods C 67. The committee accordingly asks for the necessary nine-tenths vote at the annual meeting in order that this revision, given below, may be referred to letter ballot of the Society for immediate adoption.

Section 3.—Change the fourth and fifth sentences to read as follows by the addition of the italicized words and the omission of those in brackets:

> For the purpose of tests, not less than [ten] *five* bricks shall be required for each investigation. In general, two samples of [10] *five* bricks each shall be tested for each 100,000 bricks contained in the lot under consideration; but where the total quantity exceeds 500,000, one sample of [10] *five* bricks from each 100,000, shall be tested.

IV. Adoption of Tentative Standard as Standard.—Since no criticisms or suggestions have been received for revision of the Tentative Methods of Testing Brick (Modulus of Rupture, Compressive Strength, Absorption) (C 67 – 36 T),[3] it is recommended that these tentative methods be referred to letter ballot of the Society for adoption as standard to replace the present Standard Methods of Testing Brick (Compression, Flexure, Absorption) (C 67 – 31).[4] Tentative Methods C 67 represent a clarification of the standard methods being withdrawn. Since the tentative methods were revised by the Society less than a year ago,[1] their adoption as standard requires a nine-tenths affirmative vote. The committee accordingly ask for the necessary nine-tenths vote at the annual meeting in order that these methods may be referred to letter ballot of the Society for adoption as standard.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 121.

[2] *Ibid.*, p. 123.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 780 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 432.

[4] 1936 Book of A.S.T.M. Standards, Part II, p. 140.

V. Withdrawal of Standard.—The committee recommends the withdrawal of the Standard Methods of Testing Brick (Compression, Flexure, Absorption) (C 67 – 31) since they are being replaced by the adoption as standard of the Tentative Methods C 67 – 36 T, as mentioned earlier in the report.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 38 members; the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. Proposed Tentative Standards			
Specifications for Paving Brick (C 7 – 37 T) to replace Standard Specifications C 7 – 30	26	0	2
Specifications for Sewer Brick (Made from Clay or Shale) (C 32 – 37 T) to replace Standard Specifications C 32 – 24	26	0	2
II. Proposed Revision of Tentative Standard			
Specifications for Building Brick (Made from Clay or Shale) (C 62 – 36 T)	26	1	1
III. Revision of Standards			
Specifications for Concrete Building Brick (C 55 – 34), immediate adoption	29	0	0
Specifications for Sand-Lime Building Brick (C 73 – 30), immediate adoption	29	0	0
IV. Adoption of Tentative Standard as Standard			
Methods of Testing Brick (Modulus of Rupture, Compressive Strength, Absorption) (C 67 – 36 T) to replace Standard Methods C 67 – 31	26	0	0
V. Withdrawal of Standard			
Methods of Testing Brick (Compression, Flexure, Absorption) (C 67 – 31)	26	1	1

This report has been submitted to letter ballot of the committee which consists of 38 members; 29 members returned their ballots, of whom 28 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

J. W. McBurney,
Chairman.

J. W. Whittemore,
Secretary.

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[1] August 26, 1936, through Committee E-10 on Standards.

EDITORIAL NOTE

The merger of Committee C-3 on Brick with Committee C-10 on Hollow Masonry Building Units was effected at the annual meeting by the organization of the new Committee C-15 on Manufactured Masonry Units. Subsequent to the annual meeting Committee C-15 presented to the Society on August 26, 1937, through Committee E-10 on Standards the following recommendations:

Proposed Revisions of Tentative Specifications for:
Paving Brick (C 7 – 37 T), and
Glazed Building Units (C 126 – 36 T).

The above recommendations were accepted by Committee E-10 and the specifications in their revised form appear on pp. 740 and 736, respectively.

REPORT OF COMMITTEE C-5
ON
FIRE TESTS OF MATERIALS AND CONSTRUCTION

Committee C-5 has held no meeting since its meeting on July 3, 1936, at Atlantic City, N. J., during the annual meeting of the Society.

As authorized at that meeting, the six subcommittees, which had been more or less inactive for some time, have been reorganized. A new Subcommittee VII on Size of Test Samples has been organized and assigned the following scope of work:

> (1) Inquiry into the effect of size of samples on test results; (2) what types of information can be obtained on samples smaller than the standard size for given types of construction; (3) under what conditions information on load-carrying ability can be obtained from temperature measurements without load application; and (4) the necessary distribution of thermocouples and other measuring appliances to obtain reliable results.

The subcommittee is under the chairmanship of S. H. Ingberg of the National Bureau of Standards, who reports that, while the results thus far obtained might justify the submission of proposed revisions of the present Standard Specifications for Fire Tests of Building Construction and Materials (C 19 – 33), it is deemed best to allow the matters before the subcommittee to remain under discussion for another year.

Proposed Tentative Specifications for Fire-Retardant Properties of Wood for Scaffolding and Shoring were submitted by Subcommittee VI on Fire Tests of Scaffolding (W. B. White, chairman) at the meeting on July 3, 1936, where, after discussion of the proposed specifications and the written criticisms that had been received, the specifications with certain amendments were approved for submission to letter ballot of the committee. The letter ballot resulted as follows: Out of a membership of 27, 23 members returned their ballots, of whom 17 voted affirmatively, 4 negatively, and 2 marked their ballots, "not voting."[1]

The need for standard specifications by which to judge the suitability of a material for scaffolding from the standpoint of fire resistance seems evident from a number of serious fires, originating on or in connection with scaffolding, resulting not only in the destruction of the scaffolding itself but also in extensive physical damage to buildings or structures served by them. The materials used in the construction of scaffolding and shoring are wood and steel, the former being by far the most generally used. In the case of steel, it is the framework that is made of that material, the platforms being of wood. It is self-evident that steel is not readily ignited by hot rivets, glowing cigarettes, flame torches or other light hazards to which temporary construction equipment is subject. It is wood that is the material that needs protection. In the case of a serious conflagration neither of the two materials is immune to destruction.

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[1] The classified vote on the proposed tentative specifications was as follows: Affirmative: 7 producers, 3 consumers, 7 general interests; negative: 2 producers, 2 general interests; not voting: 1 producer, 1 general interest.

No new method of testing is prescribed in the proposed specifications. The timber test described therein is one that has been in use for 33 years (devised by the late Ira H. Woolson, former chairman of Committee C-5) for official testing purposes by the building authorities of New York City in passing on the acceptability of chemically treated wood under the city building code. In effect, the proposed specifications are merely a needed yardstick for measuring the fire resistance of wood used in the construction of temporary equipment where it is deemed unnecessary to secure as great a fire resistance as required in permanent construction.

The proposed tentative specifications were submitted to Committee E-10 on Standards at its meeting on August 26, 1936, which committee deferred formal approval of the specifications pending a re-editing of the text in conformance with the Society's regulations governing the form of standards. Due to delays in editing and the preparation of a more detailed description and illustration of the furnace prescribed in the specifications, rather than resubmit them to Committee E-10 at this late date they are being presented to the Society at the annual meeting. Committee C-5 accordingly recommends that the proposed Tentative Specifications for Fire-Retardant Properties of Wood for Scaffolding and Shoring, in their edited form, as appended hereto,[1] be accepted for publication as tentative.

The committee's only existing standard, the Standard Specifications for Fire Tests of Building Construction and Materials (C 19 – 33), is apparently serving its purpose satisfactorily as no criticisms or suggestions for change have been received in the four years since it was last revised. It has found acceptance, either by reference or by incorporation, in laws, ordinances, and regulations of governmental or municipal bodies having jurisdiction over building construction.

This report has been submitted to letter ballot of the committee, which consists of 27 members; 26 members returned their ballots, of whom 24 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

RUDOLPH P. MILLER,
Chairman.

FITZHUGH TAYLOR,
Secretary.

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[1] See p. 756.

REPORT OF COMMITTEE C-8
ON
REFRACTORIES

Committee C-8 on Refractories held two meetings during the year, in Columbus, Ohio, on October 9, 1936, and in New York City on March 22, 1937, the latter in conjunction with the annual meeting of the American Ceramic Society.

The committee has been very active during the year. The use of A.S.T.M. tests and specifications pertaining to refractories is increasing in the industry.

Certain changes have been made in the organization of subcommittees and sections to increase the operating efficiency of the committee as a whole. The following subcommittees have been appointed:

I. Industrial Survey (L. C. Hewitt, chairman),
II. Research (F. A. Harvey, chairman),
III. Tests (F. A. Harvey, chairman),
IV. Heat Transfer (R. H. Heilman, chairman),
V. Precision and Tolerance (R. A. Heindl, chairman),
VI. Nomenclature (A. S. Watts, chairman),
VII. Specifications (G. A. Bole, chairman), and
VIII. Editorial (L. J. Trostel, chairman).

The following sections of Subcommittee III on Tests have been formed:

Load (C. M. Dodd, chairman)
Spalling (R. E. Birch, chairman)
Temperature (R. B. Sosman, chairman)
Slagging (C. E. Bales, chairman)
Analysis (L. J. Trostel, chairman)
Refractory Insulation (W. C. Rueckel, chairman)

Because of inactivity or completion of work, the following sections were discontinued during the year: Abrasion, Sieves, and Microstructure.

RECOMMENDATIONS AFFECTING STANDARDS

The recommendations of the committee regarding standards and tentative standards under its jurisdiction are summarized below, together with the analysis of the letter ballot on each item.

I. Proposed Tentative Standards:

Proposed Tentative Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes.—Last year[1] Committee

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 280 (1936).

C-8 withdrew the formal approval which it gave in 1932 to the modulus of rupture or flexural strength test described in the Standard Methods of Testing Brick (Compression, Flexure, Absorption) (C 67 – 31) on the grounds that this test is unsatisfactory for the testing of refractory materials. Methods C 67 were developed by Committee C-3 on Brick and are used primarily for testing paving and building brick. Committee C-8 has now prepared a procedure specifically for use in testing refractory materials and the committee accordingly recommends that the proposed Tentative Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes, as appended hereto,[1] be accepted for publication as tentative.

Proposed Tentative Methods of Chemical Analysis of Refractory Materials.—The committee recommends that these proposed tentative methods, as appended hereto,[2] be accepted for publication as tentative to replace the Standard Methods of Ultimate Chemical Analysis of Refractory Materials (C 18 – 35),[3] which are accordingly recommended to be withdrawn. The section on General Refractories of the present Standard Methods C 18, which has been standing without revision since 1921, has been replaced in the new methods by procedures for fireclay, silica, and high-alumina refractories; the section on Chrome and Chrome Ores is included in the new methods without revision, and the section on Magnesite Refactories, adopted in 1935, has been improved editorially.

Proposed Tentative Definitions of Terms Relating to Alumina-Diaspore Refractories.—For purposes of classification, the committee recommends that these proposed definitions, as appended hereto,[4] be accepted for publication as tentative. The definitions are intended to be added, when adopted, to the Standard Definitions of Terms Relating to Refractories (C 71 – 36).

II. Adoption of Tentative Standards as Standard:

The committee recommends that the following tentative method and tentative definitions be referred to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 – 36 T),[5] and

Tentative Definitions of Terms Relating to Refractories (C 71 – 36 T),[6,7] to be added to the Standard Definitions of Terms Relating to Refractories (C 71 – 36).

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[1] See p. 785.

[2] See p. 760.

[3] 1936 Book of A.S.T.M. Standards, Part II, p. 236.

[4] See p. 790.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 785 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 457.

[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 787 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 459.

[7] As a result of certain questions concerning these tentative definitions, the Advisory Committee of Committee E-8 on Nomenclature and Definitions asked that action on their adoption as standard be deferred, and at the annual meeting of the Society the definitions were accordingly continued as tentative.—ED.

III. Adoption of Tentative Revision of Standard as Standard:

The committee recommends that the tentative revision[1] of the Standard Definitions for Fireclay Refractories (C 27 – 35)[2] relating to the definition of "super duty fireclay brick," which was submitted for publication as tentative in 1935 and revised in 1936, be approved for reference to letter ballot of the Society for adoption as standard.

IV. Withdrawal of Standard:

The committee is recommending the withdrawal of the present Standard Methods of Ultimate Chemical Analysis of Refractory Materials (C 18 – 35),[3] which are being replaced by proposed Tentative Methods of Chemical Analysis of Refractory Materials, as mentioned earlier in this report.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 37 members; 32 members returned their ballots, the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. PROPOSED TENTATIVE STANDARDS			
Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes	31	0	1
Methods of Chemical Analysis of Refractory Materials	30	0	2
Definitions of Terms Relating to Alumina-Diaspore Refractories	28	0	4
II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Method of Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 – 36 T)	25	2	5
Definitions of Terms Relating to Refractories (C 71 – 36 T)[a, b]	26	5	1
III. ADOPTION OF TENTATIVE REVISION OF STANDARD AS STANDARD			
Definitions for Fireclay Refractories (C 27 – 35)	25	2	5
IV. WITHDRAWAL OF STANDARD			
Methods of Ultimate Chemical Analysis of Refractory Materials (C 18 – 35)	27	0	5

[a] The classified vote on the adoption of these Tentative Definitions was as follows: Affirmative: 13 producers, 5 consumers, 8 general interests; negative: 1 producer, 1 consumer, 3 general interests.

[b] The recommendation for the adoption as standard of these definitions failed of approval at the annual meeting of the Society.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (L. C. Hewitt, chairman).—During the year this subcommittee prepared an industrial survey pertaining to refractory service conditions in continuous bottle tank operation in the glass industry. It is planned to include this survey in the revised edition of the Refractories Manual to be issued after the annual meeting.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1198 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 1336.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 253.

[3] *Ibid.*, p. 236.

Subcommittee III on Tests (F. A. Harvey, chairman):

Section III-A on Load (C. M. Dodd, chairman) presented the proposed Tentative Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes, appended hereto,[1] which are being recommended for publication as tentative, as mentioned earlier in this report.

Section III-E on Analysis (L. J. Trostel, chairman) is responsible for the revised analytical procedures for fireclay, silica and high-alumina refractories which are being included in the proposed Tentative Methods of Chemical Analysis of Refractory Materials, appended hereto.[2]

Subcommittee IV on Heat Transfer (R. H. Heilman, chairman).—This subcommittee is working with the National Bureau of Standards on the development of a standard method for determining thermal conductivity of refractory materials at high temperatures.

Subcommittee VI on Nomenclature (A. S. Watts, chairman).—This subcommittee has prepared the definitions for three grades of alumina-diaspore refractories, which are being recommended for publication as tentative, as appended hereto.[3]

Subcommittee VII on Specifications (G. A. Bole, chairman).—This subcommittee has under discussion a proposed method of measuring warpage of special refractory shapes.

Subcommittee VIII, Editorial (L. J. Trostel, chairman).—This subcommittee has been very active in the preparation of material for the forthcoming revised edition of the Refractories Manual.[4]

This report has been submitted to letter ballot of the committee which consists of 37 members; 34 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. D. SULLIVAN,
Chairman.

E. H. VAN SCHOICK,
Vice-Chairman.

S. M. PHELPS,
Secretary.

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[1] See p. 785.
[2] See p. 760.
[3] See p. 790.
[4] Manual of A.S.T.M. Standards on Refractory Materials, November, 1937, issued as separate publication.

REPORT OF COMMITTEE C-9
ON
CONCRETE AND CONCRETE AGGREGATES

Committee C-9 on Concrete and Concrete Aggregates held three meetings during the year, in Washington, D. C., on November 21, 1936; in Chicago, Ill., on March 4, 1937, in conjunction with the spring group meetings of A.S.T.M. committees; and during the annual meeting of the Society in New York City.

During the year, Committee C-9 reviewed its field of activity and submitted for the consideration of the Executive Committee of the Society a statement of its scope in keeping with the now generally recognized fields of the several national groups which are concerned with concrete.

Subsequent to the 1936 annual meeting, Committee C-9, in cooperation with Committee D-4 on Road and Paving Materials, presented to the Society through Committee E-10 on Standards the following two proposed tentative methods of test:

Tentative Methods of:

Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 36 T), to replace the Standard Method of Test for Apparent Specific Gravity of Coarse Aggregates (D 30 – 18) and the Tentative Method of Test for Apparent Specific Gravity of Coarse Aggregates in a Saturated Condition (C 86 – 31 T).

Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 36 T), to replace the Standard Methods of Test for Apparent Specific Gravity of Sand, Stone and Slag Screenings and Other Fine Non-Bituminous Highway Materials (D 55 – 25) and the Standard Method of Test for Approximate Apparent Specific Gravity of Fine Aggregate (C 68 – 30).

These proposed methods were accepted[1] for publication as tentative on August 26, 1936, and appear in the 1936 *Proceedings*.[2] The four methods which were superseded by these two new tentative standards were accordingly discontinued.

An analysis of the present situation with respect to admixtures, made by Subcommittee XV on Admixtures (G. A. Smith, chairman), has revealed that until new information is developed little progress can be made toward the objectives of the subcommittee and consequently the subcommittee has been discontinued. It is realized, however, that admixtures are important and that with further developments Committee C-9 should undoubtedly again concern itself with matters relating to their use and coming within the scope of the committee's activities. The subject is, therefore, to be held in abeyance and not abandoned.

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[1] In submitting these tentative methods to Committee E-10 on Standards, Committee C-9 reported the following results of the letter ballot vote of a total of 33 ballots returned from a committee membership of 61: Methods C 127, affirmative 33, negative 0; Methods C 128, affirmative 32, negative 1.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 805 and 808 (1936); also 1936 Book of A.S.T.M. Tentative Standards, pp. 511 and 514.

For the development of specifications and methods of tests covering the many varieties of admixtures that have been proposed, a considerable amount of research is needed. A single item that in itself has delayed work on specifications and methods of test of many admixtures is the lack of a test for workability of concrete. Progress is being made toward the development of a test for this purpose by Subcommittee XIV on Workability of Concrete (F. R. McMillan, chairman) and by other agencies, but the difficulties are great and meanwhile it is necessary to mark time with that phase of the study of admixtures. Until the producers and users of such materials can organize research on an adequate scale, little further progress can be made by the committee.

A paper entitled "A Study of Plastic Mortar Cubes," by H. W. Leavitt, submitted by Subcommittee XI on Evaluation of Data is presented as an appendix to this report.

RECOMMENDATIONS AFFECTING STANDARDS

Committee C-9 is recommending one new tentative specification; one new tentative method of test; revisions of three tentative standards; tentative revisions of four standards; the adoption as standard of two tentative methods; and the withdrawal of two standards. These proposals are given in detail below and are listed in Table I.

I. Proposed Tentative Standards.—The committee submits for publication as tentative the following specifications and method of test:

Proposed Tentative Specifications for Lightweight Aggregate for Concrete.—In response to a need of some years' standing for a specification for lightweight aggregate a study of the requirements for such an aggregate was undertaken by Subcommittee IX on Specifications and Methods of Tests of Aggregates (H. F. Clemmer, chairman) which has resulted in the preparation of the proposed tentative specifications appended hereto.[1]

Proposed Tentative Method of Test for Abrasion of Coarse Aggregate by Use of Los Angeles Machine.—This tentative method which is appended hereto[2] was prepared in cooperation with Committee D-4 on Road and Paving Materials to meet a need for a more accelerated abrasion test which would introduce the effect of impact more decidedly.

II. Proposed Revisions of Tentative Standards.—Revisions are being proposed in three tentative standards as explained in detail below:

Tentative Specifications for Concrete Aggregates (C 33 – 36 T).[3]—It is recommended that these specifications be revised as follows, as proposed by

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[1] Additional revisions of these specifications were presented at the annual meeting, see Summary of Proceedings, p. 23. The tentative specifications appear on p. 798.

[2] See p. 801.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 788 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 482.

Subcommittee IX on Specifications and Methods of Tests of Aggregates (H. F. Clemmer, chairman), and continued as tentative:

Section 1.—Change from its present form: namely,

1. Fine aggregate shall consist of sand or other approved inert materials with similar characteristics, or a combination thereof, having hard, strong, durable particles and shall conform to the requirements of these specifications.

to read as follows:

1. Fine aggregate shall consist of natural sand, sand prepared from stone, blast-furnace slag, or gravel; or, subject to the approval of the engineer, other inert materials having similar characteristics.

Section 2.—Change Paragraph (*a*) from its present form: namely,

2. (*a*) The amount of deleterious substances shall not exceed the following maximum requirements:

	MAXIMUM PERMISSIBLE PERCENTAGE BY WEIGHT
Removed by decantation	3
Coal	1
Clay lumps	1
Other local deleterious substances (such as shale, alkali, mica, coated grains, soft and flaky particles)	..
Total coal, clay lumps, shale, soft fragments and other local deleterious substances	5

NOTE.—It is recognized that under certain conditions percentages of deleterious substances less than those shown in the table should be specified as a maximum.

to read as follows:

2. (*a*) The amount of deleterious substances shall not exceed the following limits:

	RECOMMENDED PERMISSIBLE LIMITS, PER CENT BY WEIGHT	MAXIMUM PERMISSIBLE LIMITS, PER CENT BY WEIGHT
Clay lumps, not more than	1	1.5
Coal and lignite	0.25	1
Material finer than No. 200 sieve:		
(*a*) In concrete subject to surface abrasion, not more than	2	3
(*b*) All other classes of concrete, not more than	3	5
Other deleterious substances (such as shale, alkali, mica, coated grains, soft and flaky particles)	as specified	as specified

NOTE.—The recommended limits should be specified on all work where it is economically practicable to obtain materials conforming thereto.

Section 6.—Change from its present form: namely,

6. The amount of deleterious substances shall not exceed the following maximum requirements:

	MAXIMUM PERMISSIBLE PERCENTAGE BY WEIGHT
Removed by decantation	1½
Shale (removed by flotation test)	1
Coal	1
Clay lumps	0.5
Soft fragments	5
Other local deleterious substances (such as alkali, friable, thin, elongated, or laminated pieces)	..
Total shale, coal, clay lumps and soft fragments	5

NOTE.—It is recognized that under certain conditions percentages of deleterious substances less than those shown in the table should be specified as a maximum.

to read as follows:

6. The amount of deleterious substances shall not exceed the following limits:

	RECOMMENDED PERMISSIBLE LIMITS, PER CENT BY WEIGHT	MAXIMUM PERMISSIBLE LIMITS, PER CENT BY WEIGHT
Soft fragments	2	5
Coal and lignite	0.25	1
Clay lumps	0.25	0.25
Material finer than No. 200 sieve	0.5 (Note 2)	1 (Note 2)
Other deleterious substances	as specified	as specified

NOTE 1.—The recommended requirements should be specified on all work where it is economically practicable to obtain materials conforming thereto.

NOTE 2.—When the material finer than the No. 200 sieve consists essentially of crusher dust the recommended and maximum permissible limits specified above may be raised to ¾ and 1½ per cent, respectively.

Section 7.—Add to the table of grading requirements after the ½ in. to No. 4 size a reference letter "a" to refer to the following footnote:

[a] Not more than 5 per cent passing the No. 8 sieve.

Tentative Methods of Test for Soundness of Fine Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 35 T)[1] *and for Soundness of Coarse Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 89 – 35 T).*[1]—The committee recommends that these two methods be revised and combined into a single method entitled Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate. The revised methods in their consolidated form are appended hereto.[2]

III. Revision of Standards.—The committee recommends that revisions, as given in detail below, of the following four standards be accepted for publication as tentative:

Standard Method of Making and Storing Compression Test Specimens of Concrete in the Field (C 31 – 33).[3]—The present provisions of this standard are inadequate to cover the method of storage of concrete specimens during the first 24 hr. This is especially serious now that high-early-strength cements are being used and until this matter of storage is controlled more adequately, false strength results may be obtained. The following proposed tentative revisions of this method have accordingly been prepared by Subcommittee VII on Methods and Apparatus for Testing Concrete (A. T. Goldbeck, chairman):

Section 4.—Add a new Section 4 on Storage Box to read as follows, renumbering the subsequent sections accordingly:

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, pp. 842, 837 (1935); also 1936 Book of A.S.T.M. Tentative Standards, pp. 507, 502.

[2] Additional revisions of these methods were presented at the annual meeting, see Summary of Proceedings, p. 24. The methods in their revised form appear on p. 804.

[3] 1936 Book of A.S.T.M. Standards, Part II, p. 337.

4. *Storage Box.*—In order to afford reasonably uniform temperature and moisture conditions during the first 24 hr., for curing the specimens and to protect them from damage, the cylinder molds shall be placed within a storage box (Note) so constructed and kept in such a position on the work that its air temperature when containing concrete specimens shall remain within 60 to 80 F. (16 to 27 C.). Other suitable means which provide similar temperature and moisture conditions may be used.

Note.—It is suggested that the storage box mentioned in Section 4 (*a*) be made of 1-in. dressed tongue-and-groove lumber, well braced with battens to avoid warping. The box should be well painted inside and outside and should be provided with a hinged cover and padlock.

Section 7.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and the omission of those in brackets:

7. (*a*) At the end of 24 hr., test specimens shall be removed from the [place of casting] ***storage box*** and, if desired, [molds] ***the mold*** may be removed. Those specimens intended for laboratory control tests shall be placed under moist curing conditions at approximately 70 F. and maintained therein until tested.

Standard Specifications for Curing Portland-Cement Concrete Slabs with Calcium-Chloride Admixture (C 82 – 34).[1]—On the recommendation of Subcommittee XIII on Curing of Concrete (R. W. Carlson, chairman) the following revisions in these specifications are presented as tentative:

Title.—Change to read as follows by the omission of the word in brackets:

Standard Specifications for Curing Portland-Cement Concrete [Slabs] with Calcium-Chloride Admixture.

Section 1.—Change to read as follows by the omission of the words in brackets:

1. These specifications cover the method of curing [exposed] portland-cement concrete [slabs] by accelerating the setting and hardening of the concrete by means of calcium chloride [solution] incorporated with the mixture.

Sections 3 and 4.—Replace these two sections which read as follows:

3. A concentrated solution shall be prepared by adding 100 lb. of calcium chloride to 13 gal. of water previously placed in the container. The solution shall be thoroughly stirred until it is of uniform concentration and shall have a specific gravity of from 1.31 to 1.36 at 60 F.

4. From 2 to 3 pt., according to the rate of set of the concrete, of the calcium chloride solution for each bag of cement (or for each 94 lb. of cement) shall be introduced into the drum of the mixer with the mixing water. A positive automatic mechanical method shall be used for introducing the calcium chloride solution into the mixer drum in company with the water.

by the following new Section 3, renumbering the subsequent sections accordingly:

3. *Use of Calcium Chloride.*—Calcium chloride may be added to the mix in either the dry or solution form, as follows:

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 326.

(*a*) When used in the dry form from 1 to 2 lb. of calcium chloride per bag of cement, according to the temperature[a] prevailing at the time of concreting, shall be placed in the skip with the aggregates but not in contact with the cement, just prior to discharging the contents of the skip into the mixer drum.

(*b*) When used in the solution form the following procedure shall be followed in the preparation and addition of the solution to the concrete mix: Dissolve one bag of calcium chloride (100 lb.) in a quantity of water sufficient to make 25 gal. of solution (100 qt.). The solution shall be thoroughly stirred until it is of uniform concentration. From 1 to 2 qt. of this solution per bag of cement, according to the temperature[a] prevailing at the time of concreting, shall be introduced into the drum of the mixer with the mixing water. The quantity of mixing water shall be reduced by the amount of calcium chloride solution used. A positive method shall be used for introducing the calcium chloride solution.

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[a] The following amounts are recommended: At temperatures below 80 F. (27 C.), 2 lb.; 80 to 90 F. (27 to 32 C.), 1.5 lb.; above 90 F. (32 C.), 1 lb. (1 qt. of solution contains 1 lb. of calcium chloride).

Section 5.—Change to read as follows by the addition of the italicized words and figure and the omission of the figure in brackets:

[5]. *4.* The concrete shall be finished and edged promptly after placing, as concrete containing calcium chloride hardens *more* rapidly *than plain concrete.*

Standard Specifications for Curing Portland-Cement Concrete Slabs by Surface Application of Calcium Chloride (C 83 – 36).[1]—The following minor change is also presented as tentative on the recommendation of Subcommittee XIII:

Section 4.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

4. (*a*) After removal of the burlap, *the pavement shall be sprinkled and* [2] *1.5* lb. of calcium chloride shall be *immediately* applied to each square yard of the pavement surface. The material shall be spread by means of a squeegee or suitable mechanical device, and [further shoveled or broomed if necessary] *shall be screeded or belted upon dissolution* to [secure] *insure* uniform [distribution] *coverage of the concrete slab.* [Spreading shall not be done with shovels or brooms alone.]

Standard Specifications for Ready Mixed Concrete (C 94 – 35).[2]—The following revisions prepared by Subcommittee XVIII on Ready-Mixed Concrete (R. B. Young, chairman) and amended at the meeting of Committee C-9 on March 4, 1937, are recommended for publication as tentative:

Section 14.—Change to read as follows by the omission of the words in brackets:

14. [Coarse] Aggregates shall be measured by weight. [Fine aggregates shall be measured by weight or by inundated volume in a device approved by the purchaser.] Batch weights shall be based on dry materials and shall be corrected to take into account the weight of moisture contained in the aggregates.

Section 19.—Add to the marginal heading a reference letter "a" to refer to the accompanying footnote.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 327.
[2] *Ibid.*, p. 313.

Change the sixth sentence of this section to read as follows by the addition of the italicized words and the omission of those in brackets:

> When the central mixing plant is [depended upon] *used* for the complete mixing *of concrete to be transported in an agitator truck*, the minimum mixing time for mixers of 1-cu. yd. capacity or less shall be not less than 1 min.; for larger capacities of mixers this mixing time shall be increased at the rate of 15 sec. or more for each cubic yard, or fraction thereof, additional capacity.

Immediately following the sixth sentence add a new sentence to read as follows:

> When the central mixing plant is used for the complete mixing of concrete to be transported in a non-agitating truck, the minimum mixing time for mixers of 1-cu. yd. capacity or less shall not be less than 1 min.; for larger capacities of mixers this mixing time shall be increased at the rate of 30 sec. or more for each cubic yard, or fraction thereof, additional capacity.

New Sections.—Add as new Sections 20 and 21 the seventh and eighth sentences, respectively, of Section 19, modified to read as follows, renumbering the subsequent sections accordingly:

> 20. *Agitation.*[a]—When mixed concrete is transported in a truck mixer or an agitator, the size of batch shall not exceed the rated capacity of the equipment for agitation as stated by the manufacturer, and as stamped in metal at a prominent place on the equipment. The concrete shall be agitated at the rate specified by the manufacturer as agitating speed.
>
> 21. *Shrink-Mixing.*[a]—When a truck mixer, or an agitator provided with adequate mixing blades is used for transportation, the mixing time at the central mixer may be reduced to the minimum required (about 30 sec.) to incorporate the ingredients of the mixture into a mass, and the mixing completed in the truck mixer or agitator. Under these circumstances, all ingredients for a batch shall be in the central mixer and partially mixed before any concrete is discharged to the truck mixer or agitator, and the size of batch shall not exceed the rated capacity of the equipment for shrink-mixing as stated by the manufacturer, and as stamped in metal at a prominent place on the equipment. The mixing time in the truck mixer or agitator shall be as specified for truck mixing in Section 22.

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[a] The Tentative Standards and Recommended Practices for Truck Mixers and Agitators of the Revolving Drum Type of the National Ready Mixed Concrete Association place the following limitations on Capacities and Mixing Speeds:

3. *Capacities.*—(*a*) When used for truck mixing the capacity of top-door-loading truck mixers shall be in accordance with the manufacturer's rating but the volume of mixed concrete shall not exceed 50 per cent of the gross volume of the drum.

(*b*) When used for truck mixing the capacity of end-loading truck mixers shall be in accordance with the manufacturer's rating but the volume of mixed concrete shall not exceed 57.5 per cent of the gross volume of the drum.

(*c*) When used for shrink mixing, the capacity of truck mixers shall be in accordance with the manufacturer's rating but the volume of mixed concrete shall not exceed 66.66 per cent of the gross volume of the drum.

(*d*) When used for agitating, the capacity of the truck mixer or of the truck agitator shall be in accordance with the manufacturer's rating but the volume of mixed concrete shall not exceed 80 per cent of the gross volume of the drum.

(*e*) The manufacturer shall attach to each truck mixer and truck agitator a metal plate on which is stated the capacities, in terms of volume of mixed concrete, for the various uses to which the equipment is applicable. When a capacity less than the limit indicated in Section 3 (*d*) is shown on the metal plate it indicates the maximum safe load carrying capacity for which the equipment was designed.

4. *Mixing Speeds.*—(*a*) Mixing speed shall be not less than 4 r.p.m. of the drum nor greater than a speed resulting in a peripheral velocity of the drum of 225 ft. per min. (10 to 15 r.p.m., depending on size of mixer.)

(*b*) Agitating speed shall be not less than 2 nor more than 6 r.p.m. of the drum.

Section 20.—Renumber as Section 22 and add to the marginal heading a reference letter "a" to refer to the accompanying footnote.

Section 22.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> [22] *24.* Concrete shall be delivered to the site of the work, and discharge from the hauling container shall be completed within a period of 1½ hr. (*Note 1*) after the introduction of the mixing water to the cement and aggregates, or the cement (*Note 2*) to the aggregate [when the fine aggregate contains moisture in excess of 6 per cent by weight and the coarse aggregate contains moisture in excess of 3 per cent by weight].

Immediately following this section add two new notes to read as follows:

> NOTE 1.—Research has shown that a period of 1½ hr. between the introduction of mixing water and the completion of the discharge of the batch results in no detrimental effects on the concrete; under specially favorable conditions periods up to 2 and 3 hr. may be allowed. Where air temperatures are unusually high, or the ingredients of the concrete mixture are such that an unusually quick time of set or loss of plasticity may occur, it may be necessary to specify shorter periods to insure that no damage be done to the concrete; under these circumstances an investigation, satisfactory to the engineer, with the materials to be used and in the proposed proportions, should be conducted to determine if a shorter period is necessary. It is unlikely that, except under the most unusual circumstances, it will be necessary to substitute a period of less than 1 hr. for the 1½ hr. applicable to normal conditions.
>
> NOTE 2.—The limitation on the time between the introduction of the cement to the aggregates may be waived when, in the judgment of the engineer, the aggregates are sufficiently free from moisture that there will be no harmful effects on the cement.

IV. Adoption of Tentative Standards as Standard.—The committee recommends that the following two tentative methods of tests, which have been in satisfactory use for two years, be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Method of Test for Amount of Material Finer Than No. 200 Sieve in Aggregates (*C 117 – 35 T*).[1]—This method was prepared by Committee C-9 in cooperation with Committee D-4 on Road and Paving Materials as a revision of the decantation test procedures described in the existing Standard Method of Test for Quantity of Clay and Silt in Gravel for Highway Construction (D 72 – 21)[2] and Standard Method of Decantation Test for Sand and Other Fine Aggregates (D 136 – 28).[3] With the adoption as standard of Tentative Method C 117, these two latter methods are recommended to be withdrawn.

Tentative Method of Test for Determination of Voids in Coarse Aggregate for Concrete (*Dry Rodded*) (*C 30 – 35 T*).[4]—The committee recommends that this test procedure be combined with the Standard Method of Test for

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 832 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 488.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 1050.

[3] *Ibid.*, p. 1051.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 846 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 517.

Determination of Voids in Fine Aggregate for Concrete (C 30 – 22)[1] as general methods of test for determination of voids in aggregates for concrete.

V. Withdrawal of Standards.—The committee recommends the withdrawal of the following standard methods inasmuch as they have been superseded by the Tentative Methods of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 36 T) and of Fine Aggregate (C 128 – 36 T):

Standard Method of Test for Absorption by Aggregates for Concrete (Laboratory Determinations) (C 95 – 36)[2]

Standard Method of Field Test for Absorption of Mixing Water by Aggregates for Concrete (C 96 – 36)[2]

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 60 members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. PROPOSED TENTATIVE STANDARDS			
Specifications for Lightweight Aggregate for Concrete	37	0	4
Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine	39	1	2
II. PROPOSED REVISIONS OF TENTATIVE STANDARDS			
Specifications for Concrete Aggregates (C 33 – 36 T)	40	2	2
Methods of Test for Soundness of Fine Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 35 T) and Test for Soundness of Coarse Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 89 – 35 T), revised and combined	40	1	2
III. PROPOSED TENTATIVE REVISIONS OF STANDARDS			
Method of Making and Storing Compression Test Specimens of Concrete in the Field (C 31 – 33)	42	2	0
Specifications for Curing Portland-Cement Concrete Slabs with Calcium-Chloride Admixture (C 82 – 34)[a]	33	4	7
Specifications for Curing Portland-Cement Concrete Slabs by Surface Application of Calcium Chloride (C 83 – 36)	34	3	7
Specifications for Ready Mixed Concrete (C 94 – 35)	40	1	3
IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Method of Test for Amount of Material Finer Than No. 200 Sieve in Aggregates (C 117 – 35 T), to replace Standard Methods D 72 and D 136	44	0	0
Method of Test for Determination of Voids in Coarse Aggregate for Concrete (Dry Rodded) (C 30 – 35 T)	42	0	1
V. WITHDRAWAL OF STANDARDS			
Method of Test for Absorption by Aggregates for Concrete (Laboratory Determinations) (C 95–36)	42	0	2
Method of Field Test for Absorption of Mixing Water by Aggregates for Concrete (C 96 – 36)	41	0	2

[a] The classified vote on the proposed tentative revision of the Standard Specifications for Curing Portland-Cement Concrete Slabs with Calcium-Chloride Admixture was as follows: Affirmative: 7 producers, 15 consumers, 11 general interests; negative: 0 producers, 2 consumers, 2 general interests.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 360.

[2] *Ibid.*, pp. 330 and 332.

This report has been submitted to letter ballot of the committee which consists of 60 members; 44 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. W. CRUM,
Chairman.

R. R. LITEHISER,
Secretary.

APPENDIX

A STUDY OF PLASTIC MORTAR CUBES

By H. W. LEAVITT[1]

This study, sponsored by Subcommittee XI on Evaluation of Data, of Committee C-9 on Concrete and Concrete Aggregates, is an investigation of three techniques for the preparation of 2-in. plastic mortar cubes. This report was prepared from the tests made for Subcommittee XI by ten cooperating laboratories listed later in the report.

The techniques under investigation are as follows, the details of the tests being outlined in Table I:

Series I. Molds sealed to base plates, total mixing time 2½ min.
Series II. Molds not sealed to base plates, total mixing time 2½ min.
Series III. Molds sealed to base plates, total mixing time 3½ min.

TABLE I.—OUTLINE OF TESTS.

Series	Molds	Mixing Time, min.		Number of Specimens Made Each Day	Age at Test, days	Number of Days	Total Number of Specimens
		Paste Only	Total				
I	Sealed to base plates	½	2½	6	3 and 7	60	360
II	Not sealed to base plates	½	2½	6	3 and 7	60	360
III	Sealed to base plates	1½	3½	6	3 and 7	60	360

Three cubes were fabricated for each series on 60 different days so a fully cooperating laboratory made 180 cubes for each series, or 540 cubes in all. The cubes were tested at two ages, 3 days and 7 days. Certain laboratories also presented data on tests at 28 days. The procedure followed was that described in the Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (A.S.T.M. Designation: C 109 – 34 T) of the American Society for Testing Materials,[2] except as noted.

The mixture used in all series was 1:2.77 plastic mortar with graded Ottawa sand as specified in Tentative Method C 109 – 34 T. The specimens were all 2-in. cubes made six at a time from a single batch. The three comparison specimens from a batch tested at a given age constituted a set.

For each method of test (Series I, II, and III), only six cubes were made each day, one half of which were used for tests at 3 days, and the other half for tests at 7 days. The project was planned to give reasonably adequate data.

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[1] Secretary, Maine Technology Experiment Station, University of Maine, Orono, Me.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 743 (1934); also 1937 Book of A.S.T.M. Tentative Standards, p. 459.

The following ten laboratories cooperated in the work which was started early in 1935:

Atlas Portland Cement Co.
Bessemer Limestone and Cement Co.
Bureau of Public Roads
Illinois State Highway Laboratory
Iowa State Highway Laboratory
Lehigh Portland Cement Co.
Maine State Highway Laboratory
Missouri State Highway Laboratory
National Bureau of Standards
Portland Cement Assn.

The outline of the test procedure was furnished by Mr. J. C. Pearson, Research Engineer, Lehigh Portland Cement Co.

In order that the tests made by the different laboratories can be discussed anonymously, they have been assigned designating letters, A, B, C, etc., in this report. These letter designations, however, have no relation to the order in which the laboratories are listed above. Laboratories A, F, G, H, and J, completed all of the specimens on each of the 60 days for the three series at the 3 and 7-day ages. Laboratory B failed to make any 3-day tests, but completed the 7-day and 28-day tests. Laboratory C made tests only on Series II. Laboratory D made tests only on 45-day specimens. Laboratory E made tests only on Series II and III, and Laboratory I on only Series I and II. Laboratory H ran 28-day tests.

TABLE II.—MEANS AND STANDARD DEVIATIONS OF DAILY COMPRESSIVE STRENGTHS OF MORTAR CUBES FABRICATED IN THE DIFFERENT LABORATORIES.

Laboratory	3-Day Tests						7-Day Tests					
	Mean, lb. per sq. in.			Standard Deviations, lb. per sq. in.			Mean, lb. per sq. in.			Standard Deviations, lb. per sq. in.		
	Series I	Series II	Series III	Series I	Series II	Series III	Series I	Series II	Series III	Series I	Series II	Series III
A	1446	1419	1399	61.6	65.5	72.6	2256	2210	2187	91.1	89.3	104.5
B	....	....	....	....	....	....	3135	3368	3105	181.2	198.6	175.9
C	....	1524	....	....	57.6	....	....	2182	....	....	50.1	....
D	1595	1668	1554	105.8	96.0	100.8	2768	2895	2729	138.6	125.9	163.7
E	....	1488	1418	....	63.5	80.9	....	2923	2788	....	118.0	137.2
F	1195	1222	1187	52.9	58.9	58.6	1917	1961	1909	70.9	77.5	75.9
G	1138	1126	1102	105.6	106.8	102.1	2168	2159	2091	182.1	168.0	157.7
H	1758	1827	1763	105.7	120.0	104.9	2941	3066	2939	179.4	166.2	187.6
I	1400	1466	....	83.9	89.2	....	2415	2494	....	117.5	118.4	....
J	1471	1553	1463	112.8	127.6	118.2	2489	2572	2466	171.9	197.6	150.0
Average	1429	1477	1412	89.8	87.2	90.4	2511	2583	2527	141.7	131.0	144.1

The different laboratories were allowed to choose their portland cements providing it was an ordinary commercial cement and not of the quick acting or high-early-strength type. As a result, the mortars used by the various laboratories ranged in compressive strength from 1102 to 1827 lb. per sq. in. at the 3-day age, and from 1909 to 3368 lb. per sq. in. at the 7-day age. The absolute compressive strengths between laboratories may differ significantly because of this variation in the strengths of the cements used. Unfortunately, this is not the only cause of variation in the tests as will be shown later. Each laboratory chose or made its own cube molds, which undoubtedly caused further variations in the cast specimens.[1]

MEANS AND STANDARD DEVIATIONS OF COMPRESSIVE STRENGTHS OF MORTAR CUBES ARRANGED BY TYPE OF MIX AND LABORATORY

The mean compressive strengths shown by the different types of tests as carried out by the ten cooperating laboratories are shown in Table II.

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[1] See paper by J. R. Dwyer, "Effect of Departure from Planeness of Bearing Surfaces on the Compressive Strength of Two-inch Mortar Cubes," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 351 (1936).

From the data for the 3-day tests given in Table II, two facts are evident: (1) the individual means and standard deviations of compressive strengths vary widely between laboratories and (2) the average mean strengths and standard deviations of strengths of the different series differ somewhat but do not obviously favor one type of test as being better than another. The data of the 7-day tests given in Table II support the same conclusions as have been drawn for the 3-day specimens. There is a wide variation between laboratories in the strengths of the cubes they fabricated. The differences between the means of the different series for any one laboratory are much less and do not obviously favor one type of mix over another. An accurate analysis is required to determine the significance of any differences between these series. Before proceeding in this analysis it is desirable to define or describe what is meant by a good test method.

A good test method has the following attributes: the method should involve the least work for the results obtained; the results should be easily and accurately measured; and they should be reproducible, that is, repeated tests on duplicate samples should give very nearly the same results as the original tests. The basic data for evaluating a test method is evidently the similarity or dissimilarity of the compressive strengths of test specimens made at the same and at different times by the same operator using the same methods.

TABLE III.—ANALYSIS OF VARIANCE[a] BETWEEN AND WITHIN BATCHES OF MORTAR.

Source of Variation	Degrees of Freedom	Laboratory	Mean Squares					
			3-Day Tests			7-Day Tests		
			Series I	Series II	Series III	Series I	Series II	Series III
Tests of same batch............	120	A............	1 636	4 421	2 088	5 489	14 871	7 832
		F............	1 282	1 552	1 104	2 633	3 025	3 383
		G............	1 317	1 526	1 119	4 253	4 847	4 651
		H............	12 021	8 817	10 280	31 961	42 032	33 113
		J............	3 096	3 215	2 351	10 830	10 703	7 157
Batches on successive days......	59	A............	11 188	13 281	15 611	25 307	24 269	32 085
		F............	8 712	10 634	10 833	15 720	18 896	18 876
		G............	35 311	34 718	31 770	100 657	89 554	74 548
		H............	34 311	42 498	34 342	103 638	83 548	110 113
		J............	31 729	35 751	26 817	68 040	78 952	46 289
Relative ratios,[b] *F*............	....	A............	6.84	3.00	7.48	4.61	1.63	4.21
		F............	6.80	6.85	9.81	5.97	6.25	5.58
		G............	26.81	22.75	28.39	23.67	18.48	16.03
		H............	2.85	4.82	3.34	3.24	1.99	3.33
		J............	10.25	11.12	11.41	6.28	7.38	6.47

[a] George W. Snedecor, "Calculation and Interpretation of Analysis of Variance and Covariance," Collegiate Press, Inc., Ames, Iowa, 1934, pp. 1 to 96.

Gertrude M. Cox and George W. Snedecor, "Covariance Used to Analyze the Relation Between Corn Yield and Acreage," *Journal*, Farm Economics, Vol. XVIII, No. 3, August, 1936.

[b] Ratio of larger mean square to smaller mean square: 1.2 is highly significant.

The three test specimens were made from the same mortar mix. Each series had a mix of its own, that is, there were three mixes of three specimens each on each day. The 3-day cured cubes were made from the same mix as the 7-day cured cubes. Cubes mixed on one day should be like those mixed on another day if the test is adequate. Technically, the question involved is this: do mixes on successive days vary more than random samples from a population with the variance of the individual tests? Since only five of the cooperating laboratories supplied data on all phases of this problem, the analysis will be limited to the data from these laboratories. The data, tabulated by laboratory and series, are presented in Table III.

For each of the five laboratories there are 120 possible comparisons of compressive strengths of cubes, each comparison from mortar mixed on the same day. For these laboratories there are 59 comparisons of compressive strengths on cubes made on different days. If the mixing, etc., has been uniformly performed, or can be uniformly performed, these two sets of tests should agree within reasonable limits, the limits set by the variation of the materials. These two sets of variations are isolated in Table III as, "tests of same batch" and "batches on successive days." In every case the mean square of the batches on different days is larger than that on the same day and by a highly significant amount as shown by the relative ratios, *F*. This is true for every laboratory, for the three types of mixes, and for the two curing times. No laboratory makes duplicate mixes agree. This fact is astounding and, if true for all types of mortar and cement testing, is serious.

Laboratories A and F have less variation in both the test within a batch and tests between batches than do the other laboratories. Laboratory H has a wider variation in the individual tests. Cubes in the same mix have coefficients of variation ranging from 2.0 to 6.7. The variation within mixes indicates some uniformity of tests on cubes made from the same batch of mortar.

The serious difficulty is the inability of operators to make batches alike on different days. The coefficients of variation between mixes on different days range from 7.8 to 12.2, or they have two to four times as great a variation as that between tests on the same day. The variations at the 3-day and 7-day ages are fairly constant.

TABLE IV.—ANALYSIS OF VARIANCE BETWEEN BATCHES AND LABORATORIES.

Source of Variation	Degrees of Freedom	Mean Squares					
		3-Day Tests			7-Day Tests		
		Series I	Series II	Series III	Series I	Series II	Series III
Batches	295	72.750	82 129	71 623	188 017	177 131	169 687
Laboratories	4	33 540 709	41 872 405	36 470 847	80 899 044	102 951 499	88 153 613
Relative ratio, *F*	All highly significant	461.0	509.8	509.2	430.3	581.2	519.5

Since successive batches vary in an unaccountable manner, their variation must constitute the experimental error with which laboratories should be compared. The results of the necessary analysis of variance, that is, comparing the variations due to batch differences with the variations due to laboratory differences, are shown in Table IV.

The highly significant relative ratios, *F*, found in Table IV show, what in a sense was already understood, that there are marked and highly significant differences between laboratories, even when the large experimental errors are properly taken into consideration. It is possible, therefore, to conclude that a result in one laboratory is not comparable with a result in another laboratory. Part of this difference is known to be due to the cement used.

These laboratory differences may be evaluated and the effect of differences in material in the three series largely overcome by use of proper statistical techniques.

A problem of more immediate importance, however, is a method to overcome the fact that laboratories cannot mix a batch of cement one day and have it comparable with a batch of cement mixed on the succeeding day. The study, as it was performed, offers a possibility of measuring this inaccuracy in the technique and of making due allowance for it. The reasoning behind this approach is as follows: The 3-day and 7-day test cubes were made from the same mix. If the technique of mixing from one day to the next was perfect, the test strengths of the 3-day cubes of the same mix made one day would agree with those made the following day. The same would be true for the 7-day cubes. The 3-day cubes could be used as measures of the inaccuracy of the mixing of the different batches. A method of avoiding the consequences of the variation in the daily batches and of measuring the variations of the 7-day tests as though the 3-day daily cube tests were held constant, would give the data desired, that is, the variation of the 7-day test on the assumption that the technique of making the different mixes is the same throughout the 60 daily tests. The correlation coefficients for, and the regressions of the 3-day and 7-day tests give the requisite data for this comparison. This analysis divided for the three series is presented in Table V.

TABLE V.[a]—ANALYSIS OF COVARIANCE BETWEEN 3-DAY AND 7-DAY SUMS.

Source of Variation	Degrees of Freedom	Sums of Squares and Products (X = 3 Day, Y = 7 Day)			Correlation Coefficients	Sums of Squares of Errors of Estimate
Laboratory		ΣX^2	ΣXY	ΣY^2		
SERIES I						
A	59	1 980 298	2 142 392	4 479 258	0.71933	2 161 504
F	59	1 542 085	1 078 845	2 782 498	0.52053	2 027 736
G	59	6 250 000	8 701 900	17 816 325	0.82464	5 700 635
H	59	6 073 033	6 296 667	18 343 973	0.59657	11 815 437
J	59	5 615 965	3 901 330	12 042 993	0.47439	9 332 795
Sum	295	21 461 381	22 121 134	55 465 047		31 038 107
Reduction for sum				22 801 169		
Sum of squares of errors of estimate for pooled laboratories				32 663 878		
SERIES II						
A	59	2 350 673	1 579 060	4 295 540	0.49693	3 234 810
F	59	1 882 300	751 000	3 344 593	0.29931	3 044 960
G	59	6 145 058	7 194 992	15 851 098	0.72902	7 426 782
H	59	7 522 173	4 182 207	14 788 073	0.39653	12 462 833
J	59	6 327 965	1 943 355	13 974 418	0.20666	13 377 602
Sum	295	24 228 169	15 650 614	52 253 722		39 546 987
Reduction for sum				10 109 791		
Sum of squares of errors of estimate for pooled laboratories				42 143 931		
SERIES III						
A	59	2 763 218	3 292 975	5 838 325	0.81985	1 914 029
F	59	1 917 400	1 273 600	3 340 965	0.50320	2 494 998
G	59	5 623 218	6 911 000	13 195 000	0.80231	4 701 301
H	59	6 078 493	4 688 840	19 490 060	0.75188	15 873 173
J	59	4 746 565	1 062 860	8 193 173	0.17044	7 955 175
Sum	295	21 128 894	17 229 275	50 057 523		32 938 676
Reduction for sum				14 049 383		
Sum of squares of errors of estimate for pooled laboratories				36 008 140		

[a] A detailed description of similar data may be found in the book by George W. Snedecor, "Calculation and Interpretation of Analysis of Variance and Covariance," Collegiate Press, Inc., Ames, Iowa, 1934, pp. 72 to 75.

Table V shows the data from the five laboratories making each series of tests on the 60 different days. The sums of the squared deviations from the means of the 3-day and 7-day tests together with the sums of their cross products are tabulated for each laboratory and series. The correlations between the 3-day and 7-day tests are derived from the above data together with the sum of the squares which would remain in the 7-day test if the 3-day test had been held constant. This value may be derived in several ways. In the older terminology the first value, 2,161,504 would be written as equal to:

$$N\sigma^2_{\text{7-day}}(1 - r^2)$$

where N = degrees of freedom,

$\sigma_{\text{7-day}}$ = the standard value for the 7-day tests divided by the deviation of the 7-day tests,

$(1 - r^2)$ = the 7-day reduction in variance when the 3-day test is held constant, and

r = the correlation coefficient between the 3-day and 7-day cube strengths.

The sum of squares of errors of estimate, 31,038,107 (for Series I), shows the variation which remains in the 7-day tests when the laboratory differences are eliminated through considering the results of each laboratory separately.

The summed results of the five laboratories may be treated in like manner. The sum of squares of errors of estimate for pooled laboratories is derived from the correlation between the 3-day and 7-day tests for summed data of all laboratories. This correlation is 0.6412. The variation which remains after making the 3-day tests constant is 32,663,878 and is derived for the pooled data in the same manner as for the individual laboratory data. The calculations for Series II and III are made as in Series I.

TABLE VI.—TEST FOR SIGNIFICANCE OF DIFFERENCES BETWEEN LABORATORY REGRESSIONS OF TABLE V.

Series	Source of Variation	Error of Estimate		
		Degrees of Freedom	Sum of Squares	Mean Square
I	Batches, pooled regressions	294	32 663 878	
	Batches, laboratory regressions	290	31 038 107	107 028
	Laboratory regressions	4	1 625 771	406 443
	$F = 3.80$	Significant		
II	Batches, pooled regressions	294	42 143 931	
	Batches, laboratory regressions	290	39 546 987	136 369
	Laboratory regressions	4	2 596 944	649 236
	$F = 4.76$	Significant		
III	Batches, pooled regressions	294	36 008 140	
	Batches, laboratory regressions	290	32 938 676	113 582
	Laboratory regressions	4	3 069 464	767 366
	$F = 6.76$	Significant		

From Table V we have two variances. The first variance, 31,038,107 for Series I, gives the variation within laboratories for the regression of the 7-day cube strengths on their companion 3-day cubes. The second variance, 32,663,878 for Series I, is the variance within the laboratories for the regressions of 7-day cube strengths on their comparison 3-day cubes plus the variation brought about by differences in the laboratory regressions. The two variances can be separated and a further test of laboratory differences made. Table VI presents these data.

The data of Table VI show that a significant difference between laboratory regressions exists. The change in strength from the 3-day to the 7-day period is different between the different laboratories.

Table VII adds further information concerning the analysis of the mixing, the aging, and the laboratory differences. The question here considered is whether or not the 7-day laboratory means adjusted for constant 3-day tests for all mixes would differ significantly among themselves.

The data in Table VII show that even if the 3-day tests were alike for the different cement mixes on each day in each laboratory, the adjusted 7-day laboratory means would differ markedly. The most obvious interpretation to place on such differences is that the conditions of the laboratories and the cements which they use lead to differences in the aging process. This variation is in addition to those variations which make differences in the 3-day cubes. These facts make it evident that there is no way to compare the data available from one laboratory with corresponding data from another. It will be necessary in comparing the different techniques to remain strictly within the data from each laboratory.[1] The comparisons of the different techniques as carried out by the different laboratories is presented in Table VIII.

The data in Table VIII make the two possible comparisons between the techniques, sealed *versus* unsealed molds, and $2\frac{1}{2}$ min. *versus* $3\frac{1}{2}$ min. mixing time. The results indicate that there are significant differences in these techniques as they are performed within certain of the different laboratories. When one contrasts the differences as between the laboratories, however, the significance of these differences becomes contradictory. Thus laboratory A shows a significant difference between sealed and unsealed molds in favor of a greater strength when sealed molds are used. Laboratories F, H, and J also show significant differences but in the opposite direction, a greater strength resulting from the use of the unsealed molds. The results are definitely contradictory. What evidence there is seems to favor the unsealed molds as possibly giving a slightly greater strength than the sealed molds. A mixing time of $2\frac{1}{2}$ min. seems as effective as $3\frac{1}{2}$ min. These tentative comparisons are extremely hazardous. No satisfactory conclusion with regard to these techniques can be drawn until laboratories can make their mortar mixes of any one day comparable with those made of the same materials at another time.

TABLE VII.—TEST FOR SIGNIFICANCE OF DIFFERENCES BETWEEN ADJUSTED 7-DAY LABORATORY MEANS.

Source of Variation	Degrees of Freedom	Series I		Series II		Series III	
		Sum of Squares	Mean Square	Sum of Squares	Mean Square	Sum of Squares	Mean Square
Total 7-day adjusted for 3-day	295	84 035 884	....	108 848 383	....	86 245 211	....
Batches, pooled regressions	291	32 663 878	112 247	42 143 931	144 824	36 008 140	123 739
Laboratories	4	51 372 006	12 843 002	66 704 452	16 676 113	50 237 071	12 559 268
Relative ratio, F	...	....	114.4	....	115.1	....	101.5

The comparison may be carried further by methods similar to those used earlier in this paper. If the assumption is made that the 3-day cubes are measures of the inaccuracy of the mixing of the different batches of mortar, then a method of avoiding the consequences of the variation in the daily batches and of measuring the variations of the 7-day tests as though the 3-day daily cube tests were held constant, would give the comparison needed. The constants for this comparison and those for the variation of the 7-day tests, on the assumption that the technique of making the different mixes is the same throughout the 60 daily tests, is given in Table IX. As in Table VIII, two comparisons are possible, sealed *versus* unsealed molds (Series I and II), and $2\frac{1}{2}$ min. *versus* $3\frac{1}{2}$ min. mixing time (Series I and III).

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[1] This fact was also learned by earlier studies of these same data, by means of simple correlation.

The criteria on which to judge the influence of the sealed *versus* unsealed molds or of 2½-min. *versus* 3½-min. mixing times are the ratios of the mean squares, F, at the right of Table IX. The significance of every comparison has been markedly reduced. In fact, but two are left which are significant and these could easily be expected on chance. This analysis simply reinforces the conclusions of Table VIII. The final evidence shows that as far as this plastic mortar is concerned there is no preference in the method of testing. All methods are equally good or poor. From previous statements of the requirements of a satisfactory test method, it would follow that whichever method is easiest to apply may be adopted. The methods or techniques involved in Series I, II, or III are all equally bad.

TABLE VIII.—COMPARISON OF THE DIFFERENT TECHNIQUES OF MIXING WITHIN LABORATORIES AS THEY AFFECT PLASTIC MORTAR CUBE STRENGTH.

Laboratory	Mean Squares			Mean Tests		
	Between Series I and II	Within Series I and II	Relative Ratio, F^a	Series I	Series II	Difference, Series I and II
3-DAY TESTS, SERIES I AND II						
A	62 777	12 256	5.1	1446	1419	27
F	61 754	9 686	6.4	1195	1222	−27
G	10 092	35 028	0.3	1138	1126	12
H	403 282	38 411	10.5	1758	1827	−69
J	599 359	33 747	17.7	1471	1553	−82
7-DAY TESTS, SERIES I AND II						
A	187 089	24 788	7.5	2256	2210	46
F	167 593	17 308	9.7	1917	1961	−44
G	7 340	95 106	0.1	2168	2159	9
H	1 405 987	93 593	15.0	2941	3066	−125
J	637 382	73 445	8.7	2489	2572	−83
3-DAY TESTS, SERIES I AND III						
	Between Series I and III	Within Series I and III	Relative Ratio, F^a	Series I	Series III	Difference, Series I and III
A	189 301	13 409	14.1	1446	1399	47
F	5 075	9 785	0.5	1195	1187	8
G	113 945	33 548	3.4	1138	1102	36
H	2 113	34 317	0.1	1758	1763	5
J	5 280	29 273	0.2	1471	1463	8
7-DAY TESTS, SERIES I AND III						
A	433 886	29 146	14.8	2256	2187	69
F	5 532	17 299	0.3	1917	1909	8
G	540 430	87 603	6.2	2168	2091	77
H	823	106 876	0.0	2941	2939	2
J	34 635	57 113	0.5	2489	2466	23

[a] Degrees of freedom for "between series" is 1 in each case; "within series," 118. Relative ratio, F, is 3.9 for the 5 per cent point, 6.8 for the 1 per cent point, see George W. Snedecor, "Calculation and Interpretation of Analysis of Variance and Covariance," Collegiate Press, Inc., Ames, Iowa, 1934, p. 15.

The analyses of these data have emphasized one problem. None of the laboratories studied was able to make one batch of mortar comparable with another batch, even though they used the same ingredients and performed the technique of mixing in the same manner. Is it possible to find out the cause of this serious variation?

TABLE IX.—INFLUENCE OF SEALED (SERIES I) *versus* UNSEALED MOLDS (SERIES II), AND $2\frac{1}{2}$-MIN. (SERIES I) *versus* $3\frac{1}{2}$-MIN. MIXING TIME (SERIES III) ON THE VARIATIONS OF 7-DAY TESTS (INFLUENCE OF THE 3-DAY TEST ELIMINATED).

Laboratory	Variance	Degrees of Freedom	Sums of Squares and Products (X = 3-Day, Y = 7-Day)			Variance 7-Day for Constant 3-Day	Degrees of Freedom	Mean Square	Relative Ratio, F
			ΣX^2	ΣXY	ΣY^2				
	SEALED *versus* UNSEALED MOLDS								
A	Total..................	119	1 508 992	1 350 998	3 112 022	1 902 476			
	Between Series I and II..	1	62 777	110 607	187 089				
	Within Series I and II...	118	1 446 215	1 240 391	2 924 933	1 861 074	117	15 906	
	Test of significance of differences among adjusted 7-day means..........					41 402	1	41 402	2.6
F	Total..................	119	1 204 661	712 832	2 209 957	1 788 154			
	Between Series I and II..	1	61 754	102 953	167 593				
	Within Series I and II...	118	1 142 907	610 879	2 042 364	1 715 852	117	14 665	
	Test of significance of differences among adjusted 7-day means...........					72 302	1	72 302	4.9
G	Total..................	119	4 143 355	5 308 234	11 229 814	4 429 204			
	Between Series I and II..	1	10 092	9 339	7 340				
	Within Series I and II...	118	4 133 263	5 298 895	11 222 474	4 429 244	117	37 856	
	Test of significance of differences among adjusted 7-day means..........					....	1	......	0
H	Total..................	119	4 935 733	4 246 550	12 450 001	8 796 402			
	Between Series I and II..	1	403 282	753 750	1 405 987				
	Within Series I and II...	118	4 532 451	3 492 800	11 044 014	8 352 390	117	71 388	
	Test of significance of differences among adjusted 7-day means..........					444 012	1	444 012	6.2
J	Total..................	119	4 581 553	2 566 745	9 303 852	7 865 872			
	Between Series I and II..	1	599 359	618 625	637 382				
	Within Series I and II...	118	3 982 194	1 948 120	8 666 470	7 713 488	117	65 927	
	Test of significance of differences among adjusted 7-day means..........					152 384	1	152 384	2.3
	$2\frac{1}{2}$-MIN. *versus* $3\frac{1}{2}$-MIN. MIXING TIME								
A	Total..................	119	1 771 608	2 099 198	3 873 080	1 385 717			
	Between Series I and III.	1	189 301	287 500	433 886				
	Within Series I and III..	118	1 582 307	1 811 698	3 439 194	1 364 850	117	11 665	
	Test of significance of differences among adjusted 7-day means..........					20 867	1	20 867	1.7
F	Total..................	119	1 159 661	790 117	2 046 786	1 508 452			
	Between Series I and III.	1	5 075	6 035	5 532				
	Within Series I and III..	118	1 154 586	784 082	2 041 254	1 508 782	117	12 895	
	Test of significance of differences among adjusted 7-day means..........					....	1	......	0
G	Total..................	119	4 072 583	5 453 393	10 877 538	3 575 171			
	Between Series I and III.	1	113 945	249 163	540 430				
	Within Series I and III..	118	3 958 638	5 204 230	10 337 108	3 493 796	117	29 861	
	Test of significance of differences among adjusted 7-day means..........					81 375	1	81 375	2.7
H	Total..................	119	4 051 566	3 662 692	12 612 167	9 301 029			
	Between Series I and III.	1	2 113	1 068	823				
	Within Series I and III..	118	4 049 453	3 661 624	12 611 344	9 300 405	117	79 490	
	Test of significance of differences among adjusted 7-day means..........					....	1	......	0
J	Total..................	119	3 459 460	1 668 197	6 774 024	5 969 598			
	Between Series I and III.	1	5 280	13 570	34 635				
	Within Series I and III..	118	3 454 180	1 654 627	6 739 389	5 934 963	117	50 726	
	Test of significance of differences among adjusted 7-day means..........					34 635	1	34 635	0.7

To this end, the data have been analyzed to give an estimate of the correlation between the 3-day and 7-day tests on eliminating the significant variation between the different batches of mortar. Under these conditions we obtain an answer to the question, would a superior cube in the 3-day test be superior in the 7-day test? Table X presents an analysis of the variance for all cubes divided into groups by laboratory and by series.

Table X contains enlightening data. The overwhelming contributor to the total variation in the strengths of the cubes is the difference in the 3-day and 7-day aged cubes. This was to be expected because of the different cements used by the five laboratories. The other information is not so orthodox.

TABLE X.—SOURCES OF VARIATION IN CUBE STRENGTH SEPARATED BY LABORATORY AND SERIES.

Laboratory	Source of Variation	Degrees of Freedom	Mean Square		
			Series I	Series II	Series III
A	3-day and 7-day ages	1	59 081 407	56 390 454	55 711 742
	Daily batches of mortar	59	30 351	27 696	42 903
	Age-batch interaction[a]	59	6 143	9 853	5 695
	Within the three cubes	240	3 563	9 646	4 960
F	3-day and 7-day ages	1	46 857 824	49 047 510	46 937 204
	Daily batches of mortar	59	18 312	19 009	22 049
	Age-batch interaction	59	6 121	10 522	7 661
	Within the three cubes	240	1 958	2 289	2 243
G	3-day and 7-day ages	1	95 635 562	96 058 677	88 040 774
	Daily batches of mortar	59	117 147	99 572	92 204
	Age-batch interaction	59	18 821	24 700	14 114
	Within the three cubes	240	2 785	3 186	2 885
H	3-day and 7-day ages	1	125 623 016	138 260 057	125 622 969
	Daily batches of mortar	59	104 549	86 652	98 718
	Age-batch interaction	59	33 400	39 394	45 737
	Within the three cubes	240	21 991	25 425	21 696
J	3-day and 7-day ages	1	92 710 069	93 167 405	90 530 772
	Daily batches of mortar	59	71 925	68 331	42 558
	Age-batch interaction	59	27 843	46 372	30 548
	Within the three cubes	240	6 963	6 959	4 754

[a] Interaction is a statistical term used by George W. Snedecor, "Calculation and Interpretation of Analysis of Variance and Covariance," *loc. cit.*, pp. 24, 26, and 32, to denote residual variation sometimes called experimental error.

Ideally considered, were it possible for laboratories to mix consistent batches of mortar and sample them at random in making their cubes, the mean squares for the daily batches of mortar would equal those for the three cubes made from each batch and both in turn would equal the age-batch interaction.

The variations of the daily batches of mortar (Table X) are in all cases significant when compared with the variation within the group of three cubes. The significance of these variations differs markedly between laboratories.

From Table X, the ratios obtained by dividing the mean squares for "daily batches of mortar" by the mean square for the term, "within the three cubes," for the different laboratories and series are:

Laboratory	$\text{Ratio} = \dfrac{\text{Mean Squares for “daily batches of mortar”}}{\text{Mean Squares for “within the three cubes”}}$		
	Series I	Series II	Series III
A	8.4	2.9	8.6
F	9.3	8.3	9.8
G	42.1	31.2	32.0
H	4.7	3.4	4.5
J	10.3	9.8	8.9

A ratio greater than 1.2 would not be expected in 1 to 100 trials so that we may conclude that in no case can laboratories make their mixes of mortar consistently the same on different days. Laboratory H most nearly approaches this ideal, with laboratory G the worst. However, from another aspect, laboratory H can take little pleasure in this fact because its variation within batches is too great as judged by the other laboratories.

The ratios between the "age-batch interactions" and the "within the three cubes" are:

Laboratory	Ratio = $\frac{\text{Mean Squares for "age-batch interactions"}}{\text{Mean Squares for "within three cubes"}}$		
	Series I	Series II	Series III
A	1.7	1.0	1.1
F	3.1	4.6	3.4
G	6.8	7.17	4.9
H	1.5	1.5	1.5
J	4.0	6.7	6.4

In this case a ratio of 1.2 would be significant. Laboratories A and H approach the more ideal techniques of having the interactions most nearly like the mean squares for within the group of three cubes. The values of the intraclass age-batch correlations are:

Laboratory	Ratio = $\frac{\text{Mean Squares for "age-batch interaction"}}{\text{Mean Squares for "daily batches of mortar"}}$		
	Series I	Series II	Series III
A	0.20	0.36	0.14
F	0.33	0.55	0.35
G	0.16	0.25	0.15
H	0.31	0.45	0.46
J	0.39	0.68	0.72

These correlations are consistent for the three series within laboratories, but differ rather widely between laboratories. Correlations under 0.3 may be considered rather low. Two laboratories (A and G) remain under this figure.

If we compare the mean squares for the different series and within laboratories, we note a remarkable agreement. It is consequently evident that the techniques differentiating these series have had scarcely appreciable effects. One technique of fabricating the cubes is as good as the other.

CONCLUSIONS

This paper shows that in the present state of testing plastic mortar cubes the three techniques of cube fabrication, herein discussed, lead to essentially similar test results. No one method of making cubes from plastic mortar is significantly better than another.

One of the most significant facts brought out by this study is that no one of the cooperating testing laboratories was able to make two comparable batches of plastic mortar on different days. This surprising fact is established even though each laboratory used identical materials and methods of fabrication. The extremely large variations found in the daily variations of all of the laboratories point to a pressing need for standardization of laboratory technique in testing plastic mortar, and presumably in cement testing in general.

A second source of variation found in these data in plastic mortar tests is the variation of the different laboratories. This variation is partly due to differences in the cements used (the other materials were presumably constant), and partly due to the non-standardization of the cube molds used in this study.

A third source of variation found is one which caused the laboratories to differ greatly in their ability to bring out the full possibilities of the materials entering into the plastic mortar, even when account is taken of the difference in cements.

SUGGESTIONS FOR FUTURE RESEARCH

This covariance analysis brings out many general problems in cement testing procedure. These problems may be stated as follows:

1. Why should cube molds be used to cast the test specimens?
2. Has it been conclusively proven that 2-in. cubes are definitely superior to 2 by 4-in. cylinders for the testing of plastic mortar?
3. Has it also been conclusively proven that the standard briquet is not suited for plastic mortar tests?
4. Do any of these tests measure the performance, the durability, or the probable life of concrete in field structures such as buildings, roads, and bridges?
5. Why do we require three test specimens to represent the test characteristic under examination?

In answer to the first three questions, all that can be said at the present time is that evidence is lacking to prove the relative value of these different types of test specimens.

The answer to the fourth question has not been found.

This study on plastic mortar cubes shows that the worst errors in testing do not lie in the number of specimens used to give the average. Possibly, two specimens would be just as satisfactory as three.

Improvement in the technique of making mortar batches more nearly alike should be encouraged. Specifically stated, daily mortar batches should be repeated and studied until such time as the technique can be perfected to the point where a closer agreement can be assured on the results obtained on the same material on different days.

The conclusions of this paper suggest the great need of further study of laboratory technique in the testing of plastic mortar. Such a study should have as its primary object the location of the reason for, and the remedy of, the extremely wide daily variations disclosed by this study. A parallel series of tests on standard briquets and on 2 by 4-in. cylinders should also be included. Such a study should indicate what form of test specimen is best. A further possibility would be the inclusion of the 28-day age period of curing. Some such data were submitted for this study, but time has prevented its inclusion in this paper.

REPORT OF COMMITTEE C-10
ON
HOLLOW MASONRY BUILDING UNITS

Committee C-10 on Hollow Masonry Building Units held a meeting at Washington, D. C., on March 30, 1937. This was the only meeting held since the committee presented its 1936 report to the Society at the annual meeting last year. The present membership of the committee is 29, of whom 13 are classed as producers, 4 as consumers and 12 as general interest members.

The attention of the committee during the past year has been directed almost exclusively toward three projects: (1) an investigation of methods of capping structural clay tile, (2) the development of specifications for concrete units for non-load-bearing masonry, and (3) the proposal to merge Committee C-10 and Committee C-3 on Brick.

The investigation on methods of capping is being carried out under the direction of T. R. Lawson at the Rensselaer Polytechnic Institute. It is being financed in part by the Society, through Committee E-9 on Research, in part by the donation of facilities of Rensselaer Polytechnic Institute and in part by donations of materials by manufacturers of structural clay tile. The method of capping structural clay tile required in the Standard Methods of Sampling and Testing Structural Clay Tile (C 112 – 36)[1] has been found to be convenient and satisfactory for tiles of prismatic shape but to result in relatively low values for the apparent strengths of bonding tile. Before capping bonding tile the recesses, intended for header bricks in masonry, must be filled to form units of prismatic shape. The apparent strengths of the tile are affected markedly by the properties of the material used for filling the recesses. In the current investigation, comparisons are being obtained between various types of filling for the recesses of bonding tile as well as between different materials for capping.

For several years, Subcommittee II on Concrete Units (W. G. Kaiser, chairman) has been considering specifications for concrete units for non-load-bearing masonry. A proposed specification submitted in 1932 was rejected by the committee because of a lack of technical information on the performance of concrete units in masonry. Since then several researches on the properties and performances of concrete units have been completed. The committee now recommends the publication as tentative of the proposed Specifications and Tests for Concrete Units for Non-Load-Bearing

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 183.

Masonry, appended hereto.[1] The specifications include requirements for compressive strength and moisture content at time of sampling, in addition to limits on dimensions, workmanship and finish.

In response to the suggestion of the Executive Committee of the Society, Committee C-10 considered the proposal to merge with Committee C-3 on Brick. At present, the fields of jurisdiction of the two committees are not well defined, there being several masonry products which might properly be considered as within the field of either committee. Moreover, some of the products which are of interest to both committees, such as glazed building units, double brick and other forms of masonry units, which are sometimes designated as tile and sometimes as brick, could be better defined by a joint committee on masonry units. The consumer and general interest members of Committee C-10 indicated that they were concerned with most of the products covered by both committees. A merger of the two committees would, therefore, effect a saving in their time in attending meetings. After a thorough discussion of the question, the committee voted unanimously in favor of the merger.[1] On letter ballot the vote was also favorable: out of 29 members, 25 members returned their ballots, of whom 23 voted affirmatively, and 2 marked their ballots "not voting."

RECOMMENDATIONS AFFECTING STANDARDS

Proposed Tentative Standard.—The committee recommends that the proposed Tentative Specifications and Tests for Concrete Units for Non-Load-Bearing Masonry, as appended hereto,[2] be accepted for publication as tentative. These specifications have been submitted to letter ballot of the committee which consists of 29 members; 25 members returned their ballots, of whom 20 have voted affirmatively, 2 negatively, and 3 marked their ballots "not voting."

This report has been submitted to letter ballot of the committee which consists of 29 members; 24 members returned their ballots, of whom 20 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

D. E. PARSONS,
Chairman.

F. E. EMERY,
Secretary.

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[1] The merger of Committee C-10 on Hollow Masonry Building Units with Committee C-3 on Brick was effected at the annual meeting by the organization of the new Committee C-15 on Manufactured Masonry Units.—ED.

[2] See p. 752.

REPORT OF COMMITTEE C-11
ON
GYPSUM

The meeting of Committee C-11 on Gypsum held in Washington, D. C., at the National Bureau of Standards on March 18, 1937, was the first meeting the committee has held since the meeting on March 7, 1934. Conditions were not favorable to holding meetings during the past few years. Considerable interest was shown at the spring meeting, which was well attended, and the committee contemplates a meeting in the fall to receive reports on several investigations which are being carried out by the members.

It is with a deep feeling of regret that Committee C-11 records the passing of W. H. Andrews, Supervising Chemist, Allentown Portland Cement Co.; Albin H. Beyer, Professor of Civil Engineering, Columbia University; and F. Leo Smith, Architect, American Institute of Architects, Structural Service Department, whose deaths occurred since the last report of the committee was presented in 1934.

The Standard Specifications for Sand for Use in Gypsum Plaster (C 35 – 30) and the Tentative Specifications for Sand for Use in Lime Plaster (C 66 – 31 T), the latter under the jurisdiction of Committee C-7 on Lime, have been revised and combined into a single standard entitled Tentative Specifications for Sand for Use in Plaster (C 35 – 36 T). This work was undertaken at the request of Committee E-10 on Standards by a joint conference committee representing Committees C-7 and C-11, assisted by a representative of the Sectional Committee on Specifications for Plastering (A.S.A. Project: A42). The Tentative Specifications C 35 – 36 T were submitted to the Society last year by Committee C-7 on Lime.[1] Committee C-11 concurred in this recommendation, the results of the letter ballot vote in Committee C-11 being as follows: Out of 26 members, 25 members returned their ballots, of whom 18 voted affirmatively, 1 negatively, and 6 members marked their ballots "not voting." The specifications appear in the 1936 *Proceedings*.[2] Consideration is now being given to certain proposed changes in these specifications for sand.

During the past year the Standard Specifications for Gypsum Lath (C 37 – 34), for Gypsum Wall Board (C 36 – 34) and for Gypsum Sheathing

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[1] See Report of Committee C-7 on Lime, *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 275 (1936).

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 771 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 415.

Board (C 79 – 34) have been modified by the addition to Section 9 on Finish of a note referring to the availability of gypsum lath with one surface covered with aluminum foil, and of gypsum wall board and gypsum sheathing board with one or both surfaces covered with aluminum foil. This addition was considered as an editorial change and has been incorporated in the specifications.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Gypsum Plasters (Miller Porter, chairman).—Some investigative work has been done and more is to be done on Keene's cement with a view to bringing into agreement the A.S.T.M. Specifications for Keene's Cement (C 61 – 30) and the Federal Specifications for Keene's Cement (SS-C-161). These specifications differ in several requirements, particularly as to tensile strength, fineness and testing consistency.

The determination of the time of set of gypsum neat plaster with and without standard Ottawa testing sand has been before the committee for some time. Results indicate that standard Ottawa testing sand varies decidedly in its accelerative action upon the setting time of plaster. This variation is of sufficient magnitude to render unfeasible the idea of using untreated standard Ottawa testing sand for determination of time of set of gypsum plasters; consequently Section 7 of the Standard Specifications for Gypsum Plasters (C 28 – 30) will have to be revised accordingly. This will undoubtedly be done this coming year.

Subcommittee IV on Testing Methods (C. K. Roos, chairman).—The investigative work on the ammonium acetate method of determining sand content of set plaster has been completed and it is expected that a paper on this method will be submitted to the Society for presentation at the 1938 annual meeting.

Because of the difficulty encountered in preparing satisfactory neat briquets for the determination of tensile strength, investigations have been conducted to determine the advisability of revising the specifications for gypsum to require tensile strength determinations to be made on plaster sanded 2 to 1 by weight. The results thus far obtained indicate that there is much in favor of the change.

This report has been submitted to letter ballot of the committee which consists of 24 members; 23 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. W. GINDER,
Chairman.

H. J. SCHWEIM,
Secretary.

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[1] 1936 Book of A.S.T.M. Standards, Part II, pp. 97 to 105.

REPORT OF COMMITTEE C-13
ON
CONCRETE PIPE

Committee C-13 on Concrete Pipe held one meeting during the year, in Chicago, Ill., on March 3, 1937, in addition to the meeting held in Atlantic City, N. J., on July 2, 1936, during the annual meeting of the Society.

Recommendations Affecting Standards

Adoption of Tentative Standard as Standard.—The committee recommends that the Tentative Specifications for Reinforced-Concrete Culvert Pipe (C 76 – 35 T)[1] be revised as follows and approved for submission to letter ballot of the Society for adoption as standard:

Tables I and II.—Change the headings over columns 3 and 4 in Tables I and II and over columns 6 and 7 in Table I to read as follows by the addition of the italicized word:

> Minimum Reinforcement, sq. in. per linear ft. of pipe *barrel*

In column 5 of Table I change the minimum shell thickness of 24-in. pipe from "$2\frac{5}{8}$ in." to read "$2\frac{1}{2}$ in."

Change the footnotes to Tables I and II to read as follows by the addition of the italicized sentence:

> Test loads for sand-bearing tests shall be one and one-half times those specified in this table for the three-edge-bearing tests. *It is recommended that the sand-bearing method be employed for testing 84-in. pipe.*

Section 14.—Change the second sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> If welded, [the splices and intersections] *the member at either a welded splice or intersection* shall develop a tensile strength not less than the minimum strength required for the reinforcement by the applicable specifications cited in Section 6.

Section 17.—Change the third sentence to read as follows by the addition of the italicized words:

> The crack shall be considered 0.01 in. in width when the point of the measuring gage will, *without forcing*, penetrate it $\frac{1}{16}$ in. at close intervals throughout the specified distance of 1 ft.

Section 20.—Add the following as a footnote to this section:

> It is recommended that the sand-bearing method be employed for testing 84-in. pipe.

Sections 21 and 22.—Add a footnote to read as follows, to refer to the words "2 per cent" appearing in the last sentence of Section 21 and the first sentence of Section 22:

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 817 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 467.

It is recommended that if these specifications are used for sewers, the number of pieces of pipe supplied for testing shall be in accordance with Section 19 of the Standard Specifications for Reinforced-Concrete Sewer Pipe (A.S.T.M. Designation: C 75) of the American Society for Testing Materials.

Section 26.—Change Paragraph (*b*) to read as follows by the omission of the word in brackets:

(*b*) Specimens shall be dried at a temperature not to exceed [approximately] 230 F. (110 C.) until two successive weighings at intervals of not less than 2 hr. show an increment of loss not greater than 0.1 per cent of the original weight of the specimen.

Section 33.—Change the first sentence to read as follows by the addition of the italicized words and the omission of the word in brackets:

The quality of all materials, [processes] *the process* of manufacture, and *the* finished pipe shall be subject to inspection and approval by an inspector employed by the purchaser.

This recommendation has been submitted to letter ballot of the committee which consists of 25 members; 24 members returned their ballots, of whom 22 voted affirmatively and 2 negatively.[1]

Committee C-13 has under its jurisdiction the Tentative Specifications for Concrete Irrigation Pipe (C 118 – 35 T) to which considerable study has been given during the year. It has been decided that they should be continued as tentative pending their further consideration by the committee.

This report has been submitted to letter ballot of the committee which consists of 25 members; 25 members returned their ballots, of whom 22 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

E. F. KELLEY,
Chairman.

M. W. LOVING,
Secretary.

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[1] The classified vote on this recommendation was as follows: Affirmative: 7 producers, 12 consumers, 3 general interests; negative: 2 producers.

REPORT OF COMMITTEE D-1

ON

PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held a meeting on April 15, 1937, at the Washington Duke Hotel, Durham, N. C., in conjunction with the meeting of the Paint and Varnish Division of the American Chemical Society.

During the year, on the recommendation of Committee D-1, ten specifications for pigments were submitted by the Society to the American Standards Association and approved as American standards. At the spring meeting of the committee eight additional specifications and five methods of test were approved, after a letter ballot vote of the committee, for submission by the Society to the A.S.A. for approval as American standards.

The Society is represented on the Inter-Society Color Council by four representatives, M. Rea Paul being the official delegate from Committee D-1. All A.S.T.M. standards pertaining to color will eventually be surveyed with the view to having them available to recommend as the need arises. During the past year the Council has adopted new articles of organization and procedure and has submitted to the Revision Committee of the U. S. Pharmacopoeia a means of designating colors that would be acceptable to art, science and industry, and sufficiently commonplace to be understood in a general way by the public.

Through H. A. Gardner, the American representative on an International Standards Association Committee on paint materials, the committee recommended for international acceptance the A.S.T.M. Standard Specifications for Spirits of Turpentine (D 13 – 34) and Standard Methods of Sampling and Testing Turpentine (D 233 – 36).

Committee D-1, through its various subcommittees, is studying the availability for use in specifications of laboratory methods for checking the service qualities of paint products. In order to promote still further exchange of information, the committee through its Subcommittee VII on Accelerated Tests for Protective Coatings (H. A. Nelson, chairman), is sponsoring a special symposium on "Correlation Between Accelerated Laboratory Tests and Service Tests on Protective and Decorative Coatings" at which all individuals and organizations who have been trying to use quick laboratory service tests are invited to describe and summarize the results of their work. The symposium is to be divided into three topics, as follows:[1]

Topic I. Finishes for Indoor Service (Clear and Pigmented); Leader—W. R. Fuller.
Topic II. Paints for Exterior Service on Wood; Leader—H. A. Gardner.
Topic III. Paints for Iron and Steel (Structural and Motive Service); Leader—C. F. Rassweiler.

Two new subcommittees have been organized during the past year, as follows: Subcommittee XXVII on Specifications for Interior Flat Paint under the chairmanship of E. F. Hickson, and Subcommittee XXVIII on Specifications for Spar Varnish, under the chairmanship of W. R. Fuller.

The two following subcommittees have been discontinued: Subcommittee XIV on The Preparation of Iron and Steel Surfaces for Painting and Subcommittee XXIII on Anti-Fouling Paints.

Since the last annual meeting, 16 new members have been elected, 5 resignations have been accepted and 4 deaths reported, leaving a total membership of 214.

Of the thirteen D-1 subcommittees, seven furnished reports of progress and four submitted detailed reports affecting standards that have resulted in the committee making the following recommendations to be acted upon at the annual meeting:

I. Two new tentative specifications, one tentative method of test and one tentative method of analysis are being submitted.

II. Revisions are proposed for immediate adoption in one standard method of test and two standard methods of analysis, and as tentative in five standard specifications and one standard method of analysis.

III. Revision is proposed of one tentative specification and two tentative methods of test.

IV. One tentative method is recommended for adoption as standard.

V. One tentative revision of a standard method of analysis is recommended for adoption as standard.

VI. Withdrawal is recommended of one standard method of analysis.

RECOMMENDATIONS AFFECTING STANDARDS

In this report, recommendations affecting standards and tentative standards are noted in brief form below, together with the results of the letter ballot. These recommendations are explained in the reports of the subcommittees directly responsible for them.

I. Proposed Tentative Standards.—The committee submits for publication as tentative, the following two new specifications, one method of analysis, and one method of test, as appended hereto:[2]

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[1] See Symposium on Correlation Between Accelerated Laboratory Tests and Service Tests on Protective and Decorative Coatings, December, 1937. (Available as separate publication).

[2] See pp. 842, 840, 849 and 869.

Tentative Specifications for Zinc Sulfide Magnesium Pigment, as proposed by Subcommittee XV,

Tentative Specifications for Titanium-Magnesium Pigment, as proposed by Subcommittee XV,

Tentative Methods of Routine Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow), as proposed by Subcommittee VIII, and

Tentative Method of Test for Spectral Apparent Reflectivity of Paints, to replace Standard Method D 307 – 30, as proposed by Subcommittee XVIII.

II. Proposed Revisions of Standards.—The committee recommends for immediate adoption as standard, revisions in the following methods of test and methods of analysis, and accordingly asks for the necessary nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society for immediate adoption:

Standard Methods of Test for Coarse Particles in Dry Pigments and Coarse Particles and Skins in Mixtures of Pigments and Vehicles (D 185 – 29), revised as proposed by Subcommittee VIII,

Standard Methods of Routine Analysis of Dry Red Lead (D 49 – 35), revised as proposed by Subcommittee VIII, and

Standard Methods of Routine Analysis of Titanium Pigments (D 186 – 33), revised as proposed by Subcommittee VIII.

The committee further recommends that the revisions proposed in the following specifications and method of analysis be accepted for publication as tentative:

Standard Specifications for Raw Tung Oil (D 12 – 33), as proposed by Subcommittee III,

Standard Specifications for Perilla Oil, Raw or Refined (D 125 – 23), as proposed by Subcommittee III,

Standard Method of Routine Analysis of Dry Cuprous Oxide (D 283 – 36), as proposed by Subcommittee VIII,

Standard Specifications for Zinc Oxide (D 79 – 24), as proposed by Subcommittee XV,

Standard Specifications for Basic Sulfate White Lead (D 82 – 24), as proposed by Subcommittee XV, and

Standard Specifications for Zinc Sulfide (D 386 – 36), as proposed by Subcommittee XV.

III. Revision of Tentative Standards.—The committee recommends that the following tentative specification and two methods of test be revised, as referred to in the reports of the subcommittees, and continued as tentative:

Tentative Specifications for Raw Soybean Oil (D 124 – 33 T), as proposed by Subcommittee III,

Tentative Method of Test for Comparative Hiding Power of Paints (D 344 – 32 T), as proposed by Subcommittee VIII, and

Tentative Method of Test for Comparative Hiding Power of White Pigments (D 406 – 35 T), as proposed by Subcommittee VIII.

IV. Adoption of Tentative Standard as Standard.—The committee recommends that the Tentative Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (D 333 – 34 T) be revised as indicated below and approved for reference to letter ballot of the Society for adoption as standard:

Section 6.—Omit the bending test described in this section which reads as follows:

> 6. The composition of the metal test panel for the bending test, the gage thereof and the manner of application of lacquer coating applied thereto, shall be mutually agreed upon by the buyer and seller.
>
> The test shall be made by bending the panel at a uniform rate through an angle of more than 140 deg. over a mandrel of mutually agreed upon radius.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirm-ative	Neg-ative	Ballots Marked "Not Voting"
I. PROPOSED TENTATIVE STANDARDS			
Specifications for Zinc Sulfide Magnesium Pigment[a]	52	10	26
Specifications for Titanium-Magnesium Pigment[a]	52	10	26
Methods of Routine Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow)	64	1	23
Method of Test for Spectral Apparent Reflectivity of Paints, to replace Standard Method D 307	66	0	22
II. PROPOSED REVISIONS OF STANDARDS			
Methods of Test for Coarse Particles in Dry Pigments and Coarse Particles and Skins in Mixtures of Pigments and Vehicles (D 185 – 29), immediate adoption	70	0	18
Methods of Routine Analysis of Dry Red Lead (D 49 – 35), immediate adoption	56	0	32
Methods of Routine Analysis of Titanium Pigments (D 186 – 33), immediate adoption	67	0	21
Specifications for Raw Tung Oil (D 12 – 33)	52	2	34
Specifications for Zinc Oxide (D 79 – 24)	64	0	24
Specifications for Basic Sulfate White Lead (D 82 – 24)	56	1	31
Specifications for Perilla Oil, Raw or Refined (D 125 – 23)	55	0	33
Specifications for Zinc Sulfide (D 386 – 36)	62	0	26
Method of Routine Analysis of Dry Cuprous Oxide (D 283 – 36)	50	2	36
III. REVISIONS OF TENTATIVE STANDARDS			
Specifications for Raw Soybean Oil (D 124 – 33 T)	52	0	36
Method of Test for Comparative Hiding Power of Paints (D 344 – 32 T)	72	1	15
Method of Test for Comparative Hiding Power of White Pigments (D 406 – 35 T)	72	1	15
IV. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (D 333 – 34 T), as revised	43	1	35
V. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Methods of Routine Analysis of White Linseed Oil Paints (D 215 – 35)	63	1	24
VI. WITHDRAWAL OF STANDARD			
Method of Analysis for the Color Characteristics of Paints in Terms of Fundamental Physical Units (D 307 – 30)	69	0	19

[a] The classified vote on the Tentative Specifications for Zinc Sulfide Magnesium Pigment and the Tentative Specifications for Titanium-Magnesium Pigment was as follows: Affirmative: 36 producers, 8 consumers, 8 general interests; negative: 7 producers, 2 consumers, 1 general interests.

V. Adoption as Standard of Tentative Revision of Standard.—The committee recommends that the existing tentative revision in the following standard method of analysis be referred to letter ballot of the Society for adoption as standard:

Revision of Standard Methods of Routine Analysis of White Linseed Oil Paints (D 215 – 35), as proposed by Subcommittee VIII.

VI. Withdrawal of Standard.—The committee recommends the withdrawal of the following standard which is being replaced by a new tentative method of analysis:

Standard Method of Analysis for the Color Characteristics of Paints in Terms of Fundamental Physical Units (D 307 – 30), as proposed by Subcommittee XVIII.

The above recommendations have been submitted to letter ballot of the committee which consists of 214 members; 88 members have returned their ballots. The analysis of the vote of the committee is given in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Testing of Paint Vehicles (H. A. Gardner, chairman).—In the report of Subcommittee III, appended hereto, the several recommendations mentioned earlier in this report are discussed in detail.

Subcommittee VII on Accelerated Tests for Protective Coatings (H. A. Nelson, chairman).—This subcommittee reports progress in the work of its subgroups. Group 1 on House Paints (C. H. Rose, chairman) has been engaged in comparing chalking resistance and gloss retention of a series of five paints in various accelerated weathering machines with exterior 45-deg. panels and vertical exposure weathering. A committee of three has been appointed to review the respective test data and the tentative conclusions which have been drawn. Present indications are that the accelerated weathering cycles of the machines used did not accurately predict the gloss retention and chalking of the paints on exterior vertical exposure, and that the 45-deg. tests, although presenting a truer picture than the accelerated tests, also failed to foretell loss of gloss and chalking obtained on vertical exposure. The report from Group I on these tests is given in the appended report of the subcommittee. A new test series is being considered by this group.

Group 4 on Lacquers is being organized under the chairmanship of E. H. Bucy.

Group 6 on Metal Protective Paints (J. C. Moore, chairman) has practically completed a series of tests on three paint systems applied over sand-blasted steel to determine similarity and order of failure of the accelerated tests as compared with 45-deg. and vertical weathering.

Subcommittee VIII on Methods of Analysis of Paint Materials (E. F. Hickson, chairman).—In the report of Subcommittee VIII, appended hereto, the several recommendations mentioned earlier in this report are discussed in detail.

Subcommittee IX on Varnish (W. T. Pearce, chairman).—This subcommittee reports progress in the work of its subgroups. Group 1 has completed a method for determining the reactivity of varnishes that will be placed in form for submission very shortly. Group 2 is continuing its work on the development of standard adhesion tests. Group 4 anticipates the early conclusion of its work on methods of evaluating failure of varnishes upon exposure. Group 5 reports progress in its work on checking and examining various methods for determining depths of color in varnishes. Group 6 is giving consideration to a study of the drying time of varnishes, which will involve defining of each type of drying and a study of methods for measurement.

This subcommittee has considered the present tentative methods under its jurisdiction and has decided not to recommend any changes in their status.

Subcommittee XII on Turpentine (F. P. Veitch, chairman).—This subcommittee reports progress in its work on detection of some of the newer mineral and coal-tar oil adulterants in turpentine. Consideration is also being given to the comparative results obtainable with the 3-in. immersion thermometer, as against the total-immersion Anschutz thermometer, for distillation.

Subcommittee XV on Specifications for Pigments Dry and in Oil When Marketed in That Form (H. E. Smith, chairman).—This subcommittee recommends for publication as tentative the Specifications for Zinc Sulfide Magnesium Pigment and Specifications for Titanium-Magnesium Pigment, as appended hereto.[1] These specifications cover new varieties of pigments that are attaining commercial recognition.

Revisions for publication as tentative are proposed in the Standard Specifications for Basic Sulfate White Lead (D 82 – 24)[2] in the form of separate tentative specifications appended hereto.[3] It is pointed out that the present standard specifications require a chemical composition that no longer represents the market condition for a good and acceptable pigment.

This subcommittee further recommends for publication as tentative the following revision in the Standard Specifications for Zinc Oxide (D 79 – 24).[4] Zinc oxide is now made in different varieties with respect to physical properties in order to accommodate the paint maker and it is, therefore, desirable to specify these physical properties in greater detail.

Section 3.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> 3. (*a*) The color, ***tinting strength, brightness*** and ***oil absorption*** [color strength], when specified, shall be equal to that of a sample mutually agreed upon by ***the*** buyer and ***the*** seller.

A tentative revision of a minor nature in the Standard Specifications for Zinc Sulfide (D 386 – 36)[5] is recommended, as indicated below, for the sake of simplification and to make the specifications more nearly uniform with others for similar pigments:

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[1] See pp. 842 and 840.
[2] 1936 Book of A.S.T.M. Standards, Part II, p. 604.
[3] See p. 838.
[4] 1936 Book of A.S.T.M. Standards, Part II, p. 606.
[5] *Ibid.*, p. 610.

Section 2.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> 2. (*a*) Dry Pigment.—The dry pigment shall consist of zinc sulfide, within the limits [described below, without the addition of any material other than that incident to the process of manufacture] ***prescribed in these specifications.***

This subcommittee recommends that the title of the present Standard Specifications for Zinc Sulfide Pigment (Barium) (D 385 – 36)[1] be changed to read "Standard Specifications for Zinc Sulfide - Barium Pigment," in order to avoid the use of the cumbrous parenthesis. A similar change will also be desirable in the statement of scope in Section 1. It is proposed that these modifications be considered as simply editorial changes.

This subcommittee has devoted careful study to all white pigments containing zinc and expects shortly to give attention to the desirability of proposing revisions in the Standard Specifications for Aluminum Powder for Paints (Aluminum Bronze Powder) (D 266 – 31) and Standard Specifications for Gold Bronze Powder (D 267 – 31). Consideration will also be given to the development of specifications for zinc chromate as an anticorrosion primer for steel. This subcommittee will continue the study of umbers and siennas begun two years ago, and further contemplates the possibility of modernizing and reformulating requirements for pure, unextended reds of the class covered in the Standard Specifications for Commercial Para Red (D 264 – 28).

Subcommittee XVIII on Physical Properties of Materials (M. Rea Paul, chairman).—This subcommittee recommends the withdrawal of the Standard Method of Analysis for the Color Characteristics of Paints in Terms of Fundamental Physical Units (D 307 – 30),[2] and the publication as tentative, of a complete revision of this standard method under the new title of "Method of Test for Spectral Apparent Reflectivity of Paints," as appended hereto.[3] Advancement in the science of colorimetrics has rendered obsolete some of the requirements set forth in the present Standard Method D 307, which made necessary the preparation of a new tentative method which includes a number of improvements and brings this test up to date.

This subcommittee has developed several definitions in connection with gloss and its specification that should be in form for presentation in the near future.

Subcommittee XXV on Cellulose Ester Coatings (R. M. Carter, chairman).—This subcommittee recommends that the Tentative Methods of

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 612.
[2] *Ibid.*, p. 822.
[3] See p. 869.

Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (D 333 - 34 T) be adopted as standard with the deletion of the bend test as described earlier in this report.

This subcommittee reports marked increase of interest in work on solvents and finished lacquers.

Subcommittee XXVI on Underground Pipe Protection (F. N. Speller, chairman).—This subcommittee has obtained considerable data regarding impact tests and durability of bituminous primers under alternate immersion and exposure to weather. The subcommittee believes that two specifications, one for coal-tar primers and the other for asphalt-base primers, might prove sufficient. Work on the development of these specifications will be delayed pending receipt of further information on adhesion and durability of these materials. The subcommittee requests that coating manufacturers bring to the committee's attention any specifications for bituminous primers for cold application to iron and steel pipes, together with suggestions on the preparation of surfaces and data on performance tests.

This report has been submitted to letter ballot of the committee which consists of 214 members; 88 members returned their ballots, of whom 71 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

ALLEN ROGERS,
Chairman.

M. REA PAUL,
Secretary.

REPORT OF SUBCOMMITTEE III ON TESTING OF PAINT VEHICLES

Subcommittee III on Testing of Paint Vehicles is recommending tentative revisions in two standard specifications and revisions in one tentative specification, as follows:

Standard Specifications for Perilla Oil, Raw or Refined (D 125 – 23):[1]

Section 1.—Change the requirement for color of perilla oil to read as follows by the addition of the italicized words:

Color.................................Not darker than a freshly prepared solution of 1.0 g. potassium dichromate in 100 cc. pure H_2SO_4 (sp. gr. 1.84), ***or its equivalent in iron-cobalt solution, or in Lovibond glasses.***

Add to Section 3 the following requirement:

Appearance.......................................clear and transparent at 65 C.

Section 3 (h).—Change the test for determining color of perilla oil from its present form to read as follows:

(*h*) *Color.*—Prepare a fresh solution of pure potassium bichromate in pure colorless H_2SO_4 (sp. gr. 1.84). This solution should be in the proportion of 1.0 g. of potassium bichromate to 100 ml. (184.0 g.) of H_2SO_4. Place the oil and colored solution in separate thin-walled, clear glass tubes of the same diameter (1 to 2 cm.) to a depth of not less than 2.5 cm. and compare the depths of color by looking transversely through the columns of liquid by transmitted light.

The iron-cobalt liquid equivalent to the above bichromate solution may be prepared as follows: Mix 29.4 ml. of Solution No. 1 with 17.6 ml. of Solution No. 2 and 53.0 ml. of Solution No. 3. The three base solutions referred to have the following composition:

Solution No. 1	$FeCl_3 \cdot 6H_2O$	5.0 g.
	HCl (2 per cent)	1.2 g.
Solution No. 2	$CoCl_2 \cdot 6H_2O$	1.0 g.
	HCl (2 per cent)	3.0 g.
Solution No. 3	Aqueous solution of HCl (2 per cent)	

The Lovibond equivalent to the above iron-cobalt solution shall be as follows: red 17.5, yellow 120.0, and blue 0.20.

The depth of liquid shall be 10.5 mm.

Add the following test for appearance of perilla oil to this section as a new Paragraph (*i*):

(*i*) *Appearance.*—If the oil is cloudy when cold, it shall become clear and transparent when heated at 65 C. for 5 min.

Standard Specifications for Raw Tung Oil (D 12 – 33):[2]

Section 1.—Change the table in this section by the addition of the following requirements for color and appearance of raw tung oil:

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 729.

[2] *Ibid.*, p. 734.

Color	Not darker than a freshly prepared solution of 1.0 g. potassium dichromate in 100 ml. pure H_2SO_4 (sp. gr. 1.84), or its equivalent in iron-cobalt solution, or in Lovibond glasses.
Appearance	clear and transparent at 65 C.

Section 3.—Add the following note at the end of Paragraph (*c*) of this section:

NOTE.—If tung oil of low acid value and pale color is desired, the maximum acid value should be reduced to 3.0, and the maximum color to 0.03 g. of dichromate per 100 ml. of H_2SO_4 (or iron-cobalt, or Lovibond equivalents).

Add to this section as a new Paragraph (*j*) a description of the test for determining color identical with that recommended above for inclusion in Section 3 (*h*) of Specifications D 125 – 23. At the end of this new Paragraph (*j*) add the same Note as proposed above for inclusion in Paragraph (*c*).

Add to this section as a new Paragraph (*k*) a description of the test for appearance identical with that recommended above in Section 3 (*i*) of Specifications D 125 – 23.

Tentative Specifications for Raw Soybean Oil (D 124 – 33 T):[1]

Section 1.—Revise the table in this section by changing the present requirement for loss on heating at 105 to 110 C. from "0.1 max., per cent" to read "0.2 max., per cent."

After the requirement covering saponification number in this table, insert the following requirements:

Unsaponifiable matter, per cent	1.5 max.
Appearance	clear and transparent at 65 C.

Add at the end of the requirement for color, appearing in this table, the phrase "or in Lovibond glasses."

Section 3.—Add the following test for unsaponifiable matter of raw soybean oil as a new Paragraph (*g*), relettering the remaining paragraphs accordingly:

(*g*) *Unsaponifiable Matter.*—Weigh 8 to 10 g. of the oil. Transfer to a 250-ml. long-neck flask. Add 5 ml. of a strong solution of NaOH (1:1) and 50 ml. of ethyl alcohol (95 per cent). Put a condenser loop inside the neck of the flask and boil for 2 hr. Occasionally agitate the flask to break up the liquid, but do not project the liquid onto the sides of the flask. At the end of 2 hr., remove the condenser and allow the liquid to boil down to about 25 ml.

Transfer to a 500-ml. glass-stoppered separatory funnel, rinsing with water. Dilute with water to 250 ml., and add 100 ml. of redistilled ether. Stopper and shake for 1 min. Let stand until the separation between the two layers is sharp and clear. Draw off all but one or two drops of the aqueous layer into a second 500-ml. separatory funnel and repeat the process, using 60 ml. of ether. After thorough separation, draw off the aqueous solution into a 400-ml. beaker, then transfer the ether solution to the first separatory funnel, rinsing down with a little water. Return the aqueous solution to the second separatory funnel and shake out again with 60 ml. of ether in a similar manner, finally drawing the aqueous solution into the beaker and rinsing the ether into the first separatory funnel.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 720 (1933); also 1936 Book of A.S.T.M. Tentative Standards, p. 583.

Shake the combined ether solution with the combined water rinsings and let stand until the separation between the two layers is sharp and clear. Draw off the water and add it to the main aqueous solution. Shake the ether solution with two portions of water (about 25 ml. each). Add these to the main water solution.

Swirl the separatory funnel so as to bring the last drops of water down to the stopcock and draw off until the ether solution just fills the bore of the stopcock. Wipe out the stem of the separatory funnel with a bit of cotton on a wire. Draw off the ether solution (portionwise if necessary) into a 250-ml. flask and distill off. While still hot, drain the flask into a small weighed beaker, rinsing with a little ether. Evaporate this ether, cool the beaker, and weigh.

NOTE.—The unsaponifiable oil from adulterated drying oils may be volatile and as a consequence may evaporate on long heating. Therefore, heat the beaker on a warm plate, occasionally blowing out with a current of dry air. Discontinue heating as soon as the odor of ether is gone.

Reletter Paragraph (*h*) on color as Paragraph (*i*) and make the description of the test for color identical with that recommended above in Section 3 (*h*) of Specifications D 125 – 23.

Add to this section as a new Paragraph (*j*) a description of the test for appearance identical with that recommended above in Section 3 (*i*) of Specifications D 125 – 23.

The subcommittee is considering the preparation of specifications for such oils as oiticica, hempseed, sunflower and menhaden. Preliminary work toward establishing specifications for these oils will be carried on during the coming year.

Respectfully submitted on behalf of the subcommittee,

H. A. GARDNER,
Chairman.

REPORT OF SUBCOMMITTEE VII ON ACCELERATED TESTS FOR PROTECTIVE COATINGS

Group I on House Paints (C. H. Rose, chairman) of Subcommittee VII, has been engaged in studying the chalking and gloss retention of a series of five house paints under various weathering conditions.

Eleven accelerated tests have been made on these paints. Fifteen exterior 45-deg. exposure tests and fourteen vertical exposure tests have been made in twelve locations. The various cooperators submitted a record of their inspections and also evaluated the results of the exposure tests.

This report from Group I is based upon the findings of the Review Committee which studied the results reported by the individual cooperators.

At the April 15, 1937, meeting of Group I at Chapel Hill, N. C., a committee was appointed to review the replies of the cooperators to the questionnaire about the 1935-1936 paint tests which was designed to show the correlation of the laboratory accelerated tests and the 45-deg. tests with the vertical exposure tests for gloss retention and chalking. The Review Committee comprised Messrs. F. C. Schmutz, J. F. Broeker, and C. H. Rose.

The Review Committee has studied the test data and replies to the questionnaire and in a few cases believe that the cooperators' data lead to a different answer to Questions A and B.

Question A. Having only the accelerated test data, would the south exterior vertical exposures be correctly predicted?

Question B. Having only the 45-deg. test data, would the south exterior vertical exposures be correctly predicted?

A summary of the replies on Questions A and B is presented in the following tabulation:

	Question A			Question B		
	Exactly	Approximately	Not At All	Exactly	Approximately	Not At All
Cooperators' Conclusions..	None	3	5	2	5	4
Review Committee's Conclusions..........	1[a]	4[b]	4	2[c]	7[d]	2
Review Committee's Suggestions for Final Report	None	4	5	1	5	5

[a] In the case of this reply there was some question as to whether the agreement was exact or approximate.

[b] In the case of one reply there was some question as to whether the agreement was approximate or no agreement.

[c] In the case of one reply there was some question as to whether the agreement was exact or approximate.

[d] In the case of three replies there was some question as to whether the agreement was approximate or not at all.

The suggestion for the final report was made on a basis of reporting the doubtful agreement as being in the next class.

In the accelerated weathering tests, machines with carbon-arc light and mercury-vapor-lamp light were used. The correlation of the accelerated tests with the vertical exposure tests was as follows:

	REVIEW COMMITTEE'S CONCLUSIONS	COOPERATOR'S CONCLUSIONS
Machines with Carbon Arc (Five):		
Exact correlation with exterior vertical exposure tests	none	none
Exact correlation with exterior vertical exposure tests, but with a question as to whether correlation was exact or approximate	1	none
Approximate correlation	1	1
No correlation	3	3
Machines with Mercury-Vapor Lamp (Three):		
Exact correlation with exterior vertical exposure tests	none	none
Approximate correlation	2[a]	2[a]
Approximate correlation but with a question as to whether correlation was approximate or no correlation	1	none
No correlation	1	2

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[a] Two tests in the same machine.

Correlation of Accelerated Tests with South Exterior Vertical Exposure Tests:

Data on nine accelerated tests and corresponding vertical exposure tests are available. In the opinion of Group I none of the accelerated tests exactly predicted the exterior exposure in regard to chalking and gloss retention; four accelerated tests gave an approximately correct prediction, and five accelerated tests gave a false prediction.

Correlation of 45-deg. Exposure Tests with South Vertical Exposure Tests:

Data on eleven exterior 45-deg. exposure tests and corresponding exterior vertical exposure tests are available. In the opinion of Group I one 45-deg. test gave an exact prediction in regard to chalking and gloss retention, five tests gave an approximately correct prediction and five tests gave a false prediction.

The accelerated weathering tests used in this study were the ones regularly used by the individual cooperators. The exterior vertical exposure tests show that none of the accelerated weathering procedures used could be recommended by Subcommittee VII as being entirely suitable for testing house paints for gloss and chalking. Although the 45-deg. tests presented a truer picture of the ultimate failure of the paints in respect to chalking and gloss retention than the accelerated tests, the 45-deg. tests failed to satisfactorily foretell the loss of gloss or the chalking obtained on vertical exposure.

Light Source, Accelerated Weathering Machines:

Five accelerated weathering machines had carbon arcs as the light source. In the opinion of Group I two machines gave results which were approximately the same as the exterior exposure results and three machines gave results which did not correlate with the exterior tests.

Four accelerated weathering tests were made in machines with mercury-vapor lamps, there were three machines, two tests being made in the same machine, and in the case of this machine the two accelerated tests gave approximate correlation with the exterior results. The two other machines with the mercury-vapor lamp gave no correlation.

Conclusions:

These tests indicate a need for further study of accelerated weathering and the development of an accelerated weathering procedure or cycle which will more closely foretell the failure of house paints in respect to gloss and chalking and also other types of failure when exposed to exterior conditions.

Respectfully submitted on behalf of the subcommittee,

H. A. NELSON,
Chairman.

REPORT OF SUBCOMMITTEE VIII ON METHODS OF ANALYSIS OF PAINT MATERIALS

Methods of Analysis:

The Society has no methods for the analysis of zinc yellow. This rather old pigment has come into prominence in recent years as a constituent of priming paints for metal. The U. S. Navy uses a great deal of this pigment in aircraft paint and the National Bureau of Standards is receiving an increasing number of paints for priming metal, based on a zinc yellow-iron oxide combination. At the request of the Bureau of Construction and Repair of the U. S. Navy, Subcommittee XV is preparing specifications for zinc yellow. Thus the need of A.S.T.M. methods of analysis for this pigment is apparent and Subcommittee VIII has accordingly prepared proposed Tentative Methods of Routine Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow), which it is recommending for publication as tentative, as appended hereto.[1]

Recommendation is made for the advancement to standard of the present tentative revision[2] of the Standard Methods of Routine Analysis of White Linseed Oil Paints (D 215 – 35).[3]

This subcommittee recommends for immediate adoption the following revision in the Standard Methods of Test for Coarse Particles in Dry Pigments and Coarse Particles and Skins in Mixtures of Pigments and Vehicles (D 185 – 29),[4] since it has been suggested by several paint technologists that a No. 325 sieve test is of no value on enamels. For example, in the latest revision of the Federal Specifications for Black Enamel (TT-E-521) and for Red Enamel (TT-E-531a) the sieve test has been omitted as being of no value.

Procedure B.—Change the title of Procedure B to read as follows by the addition of the italicized word and the omission of the words in brackets:

> B. Water-Soluble Pigments, Pastes in Oil, Pastes in Japan, *and* Mixed Paints [and Enamels].

This subcommittee likewise recommends for immediate adoption the following revision in the Standard Methods of Routine Analysis of Titanium Pigments (D 186 – 33)[5] since it feels it is more desirable to refer to the standard method of test for tinting strength than to have a separate method of test for tinting strength of titanium pigments, as is the case at present:

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[1] See p. 849.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1201 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 1339.

[3] 1936 Book of A.S.T.M. Standards, Part II, p. 662.

[4] *Ibid.*, p. 621.

[5] *Ibid.*, p. 656.

Section 4.—Change this section, which describes a test for tinting strength, from its present form to read as follows:

> 4. Tinting strength shall be determined in accordance with the Standard Method of Test for Tinting Strength of White Pigments or White Pigment Pastes (A.S.T.M. Designation: D 332) of the American Society for Testing Materials.

The use of the chloroform-acetic acid wetting agent for red lead that has been extracted from a paste-in-oil or paint was incorporated in the Methods of Routine Analysis of Dry Red Lead (D 49 – 35) about 1928. It was not intended to be applied to *dry* red lead; however, it was written to include both dry powders and extracted pigments. There is no need of the wetting agent with the dry powder and there is also the danger that an error in analysis might be due to its use, particularly in the hands of an inexperienced analyst. Its use in extracted pigments (that may contain a small amount of unextracted oil) is desirable. Thus Subcommittee VIII is recommending for immediate adoption the following revision in Standard Methods D 49:[1]

Section 8.—Change the first two sentences from their present form to read as follows:

> Weigh 1 g. of the finely-ground sample, transfer to a 200-ml. Erlenmeyer flask, add 20 ml. of distilled water; then add as quickly as possible 40 ml. of the "red-lead solution" at room temperature. If the sample is red-lead which has been extracted from the paint or paste, in place of the distilled water use 10 ml. of a mixture of 7 parts by volume of chloroform and 3 parts by volume of c.p. glacial acetic acid; and then add without delay the red-lead solution. Add 30 ml. of water containing 5 or 6 g. of sodium acetate and titrate at once with 0.1 *N* sodium thiosulfate, adding the latter rather slowly and keeping the liquid constantly in motion by whirling the flask.

This subcommittee further recommends that the following editorial change be made immediately in the Standard Methods of Test for Specific Gravity of Pigments (D 153 – 27):[2]

Section 14.—Delete the illustration identified as Fig. 4, and substitute the drawing shown in the accompanying Fig. 1. This change is presented in order to correct the illustration of this apparatus and does not affect the accuracy of the method.

The present procedure for determining cuprous oxide in the Standard Method of Routine Analysis of Dry Cuprous Oxide (D 283 – 36)[3] includes any metallic copper present and although attention is called to this in the method, it is now considered important to correct the determination for the metallic copper present since cuprous oxide is the important ingredient for toxicity in antifouling paints. A study was accordingly undertaken of the method proposed by Hurd and Clark.[4] The results of these tests are quite satisfactory and the subcommittee is accordingly recommending that the completely revised method appended hereto[1] be published as a tentative revision of the existing Standard D 283 – 36.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 633.

[2] *Ibid.*, p. 638.

[3] *Ibid.*, p. 624.

[4] L. C. Hurd and A. R. Clark, "Determination of Metallic Copper in Cuprous Oxide - Cupric Oxide Mixtures," *Industrial and Engineering Chemistry* Analytical Edition, Vol. 8, No. 5, p. 380 (1936).

Tests for Relative Dry Hiding Power of Paints and Pigments:

The subcommittee is also recommending extensive revisions of the Tentative Methods of Test for Comparative Hiding Power of Paints (D 344 – 32 T) and Test for Comparative Hiding Power of White Pigments (D 406 – 35 T), as appended hereto,[2] entitled, Tentative Methods of Test for Relative Dry Hiding Power of Paints, and Test for Relative Dry Hiding

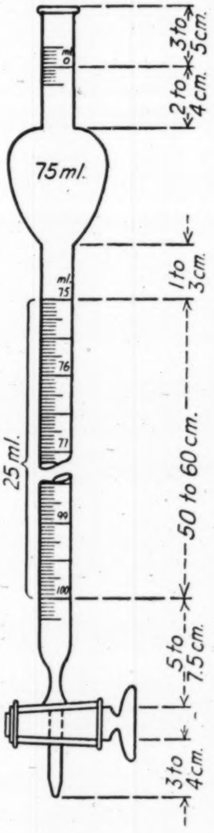

FIG. 1.—Glass Burette.

Burette: Geissler, straight; glass stopcock, ground accurately.

Total capacity	100 ml.	Tolerances:	
Capacity of bulb	0 to 75 ml.	Total capacity	±0.10 ml.
Graduated	75 to 100 ml.	Graduated portion	±0.03 ml.
Subdivisions	0.05 ml.		
Rate of outflow	about 2 min.		

Markings on graduations should be in conformity with the Bureau of Standards Circular No. 9

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[1] See p. 855.

[2] See pp. 866 and 844.

Power of White Pigments. There is given below a detailed report of the studies carried out by the work committees responsible for the development of these revised hiding power methods:

Hiding Power of Paints:

In May, 1935, G. F. A. Stutz, who was then chairman of Subcommittee VIII, appointed a Hiding Power Work Committee consisting of D. L. Gamble, chairman, R. H. Sawyer, and A. E. Jacobsen. The purpose of this committee was to consider various proposed methods of making brushout hiding power determinations on paints, to recommend to the subcommittee the method considered most feasible, and to work out the details of the method.

At the meeting of the subcommittee held on June 24, 1935, the work committee submitted a report dealing with the various methods for brushout hiding power and recommending the adoption of a dry film, incomplete hiding test. As a result, the subcommittee instructed the work committee to proceed with the development of a specific detailed method for determining hiding power which would include the use of a standard paint and both white and black and white and gray backgrounds, the measurements to be made on dry films at incomplete hiding.

Design of Brushout Board, Contrast.—At the Detroit meeting the subcommittee expressed a preference for an initial white and gray (40 per cent brightness) contrast but suggested also the use of the black and white as an additional brushout board which would permit the method to be used on the highest hiding white paints as well as tinted and colored paints. In the experience of the members of the work committee, the relative hiding power ratings of paints, as determined by incomplete hiding tests with a board having initial black and white contrast as compared with ratings obtained with a board having an initial contrast consisting of white and gray of 40 per cent brightness, differ so little as to fall within the reproducibility of the test method. This fact, in addition to the necessity of controlling the brightness of the gray portion of the board within relatively narrow limits, led the work committee to recommend dropping the gray and white board and to standardize on the black and white board.

Brightness Tolerances.—Work was carried out by the committee to determine the permissible brightness tolerances in the contrasting areas of the board. The established tolerances are stated in Section 3 (*e*) of the proposed revised method appended hereto.[1]

Geometrical Design.—As a result of work with various geometrical designs, the members of the work committee indicated a preference for a design consisting of alternate diagonal ½-in. black-and-white stripes. It is felt that this design aids somewhat in obtaining uniform application of the paint and tends to make grading of the brushouts easier. However, the committee does not feel that the results to be obtained with the proposed test method are sufficiently critical in respect to the geometrical design of the board to necessitate specifying an exact design.

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[1] See p. 866.

In this connection, work was carried out with a board consisting of a diamond design and having located in juxtaposition at the center a black and a white square, each about 3-in. on a side. The purpose of this design was to permit photometric measurements if desired. As will be seen from results of a cooperative test to be given later in this report, the results obtained with this design were as satisfactory as those obtained in the case of the usual diamond pattern. However, two of the four operators cooperating in the tests reported that the central squares made grading more difficult.

Definition of Hiding Power.—Since the hiding power as measured by an incomplete hiding test, such as is being proposed, does not fit the present definition as given in the Standard Definitions of Terms Relating to Paint Specifications (D 16 – 24), a new definition for relative dry hiding power of a paint has been drawn up by the committee and is included in Section 2 of the proposed method.

Paint	Relative Dry Hiding Power of Paint Determined by Proposed Method							
	Brushout Board No. 1				Brushout Board No. 2			
	Operator				Operator			
	1	2	3	4	1	2	3	4
No. 1 Gloss (Standard)	100	100	100	100	100	100	100	100
No. 2 Gloss	139	139.9	138	...	132	141.2	137	136
No. 1 Flat (Standard)	100	100	100	100	100	100	100	100
No. 2 Flat	124	124.1	121	122.3	125	123.8	122	121.3

Cooperative Tests.—In the test method being proposed, a general procedure is given in which a paint can be rated as equal to, better than, or inferior to a standard paint. In many cases it is not sufficient to say the sample paint is better or poorer than the standard and the amount of difference must be determined quantitatively. For this reason, an optional procedure has been included in the test method which permits quantitative determination of the relative dry hiding power of a paint in terms of a standard paint as defined above. Given in the tabulation above are results of cooperative tests carried out by the three committee members and E. F. Hickson of the Bureau of Standards using the proposed optional procedure. These were carried out on two gloss and two flat paints using two different brushout boards. Brushout Board No. 1 consisted of a diamond pattern with ½-in. black-and-white stripes while board No. 2 was of a similar design but having at its center a black and a white square to permit photometric measurements.

In the case of gloss paint No. 2 with seven determinations obtained by four operators using two brushout boards of different design, the maximum deviation from the average is 4 per cent while in the case of flat paint No. 2 the maximum deviation from the average is less than 2 per cent.

Hiding Power of Pigments:

The work committee in reporting the new method of test for the hiding power of paints, appended hereto,[1] also recommended that the Tentative Method for Hiding Power of White Pigments (D 406 – 35 T) should be revised to conform with the new method of test for paints.

A second work committee, consisting of D. L. Gamble, chairman, A. E. Jacobsen, R. H. Sawyer, and G. G. Sward, was instructed by Subcommittee VIII to proceed with this revision.

Method of Hiding Power Measurement.—The dry film hiding power test method proposed in the new methods of test for paint has been carried over essentially without modification into the proposed new method of test for relative dry hiding power of white pigments appended hereto.[2]

Method of Preparing a Paint of the Pigment to be Used.—The method of preparing paints from the pigments to be tested has been modified only slightly from that specified in the present Tentative Method D 406 – 35 T with the exception that the refined linseed oil used in the present method has been replaced by a vehicle consisting of a mixture of heat-bodied alkaline refined linseed oil and light boiled linseed oil to which is added cobalt naphthenate drier in order to obtain a simple paint without thinner, but possessing drying properties which will permit hiding power measurements on a basis of a dry hiding power test. The table which appears in Section 5 (*a*) of the present tentative method has been revised and is included in the proposed revised method to serve as a guide in using the test method.

Definition of Hiding Power of White Pigment.—Since hiding power as measured by an incomplete hiding test, such as is being proposed, does not fit the present definition as given in the Standard Definitions of Terms Relating to Paint Specifications (D 16 – 24), a definition of the relative dry hiding power of a white pigment has been drawn up by the committee and is included in Section 2 of the proposed method.

Cooperative Tests.—In order to test the over-all reproducibility of the method of test being proposed, cooperative tests were carried out by members of the work committee, including E. F. Hickson. Samples of titanium dioxide and zinc sulfide – barium pigment were submitted to the members and each was tested by the proposed method against itself. In addition, samples of high-tinting-strength white lead and ordinary Carter process white lead were submitted to each member and the hiding power of the Carter process white lead was determined by the new proposed method using the high-tinting-strength material as a standard pigment. The two white-lead pigments were selected for this purpose because they represent two slightly different pigments of the same general type. It was felt that this latter test would yield information indicative of the uniformity of grind obtainable by the proposed mulling procedure from laboratory to laboratory. In carrying out these tests the optional procedure included in the proposed test method was used. This was done in order that these cooperative tests would yield quantitative data.

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[1] See p. 866.

[2] See p. 844.

The following tabulation gives the results of the cooperative tests:

Relative Dry Hiding Power of White Pigments

Pigment	Standard	Operator			
		1	2	3	4
Titanium dioxide	Titanium dioxide	95	99.4	97.2	....
Zinc sulfide - barium	Zinc sulfide - barium	98	100.7	97	....
Carter white lead	High-tinting-strength white lead	82	80.2	80.8	83.4

The over-all reproducibility of the test method is indicated to be of the order of magnitude of 5 per cent. Although this degree of reproducibility may not be all that is desired, it is definitely better than that obtained in cooperative tinting strength tests based on the present Standard Method for the Tinting Strength of White Pigments or White Pigment Pastes (D 332 - 36).[1]

Respectfully submitted on behalf of the subcommittee,

E. F. Hickson,
Chairman.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 647.

REPORT OF COMMITTEE D-2

ON

PETROLEUM PRODUCTS AND LUBRICANTS

Committee D-2 on Petroleum Products and Lubricants held three regular meetings during the past year (in June at Atlantic City, N. J., in January at New York, N. Y., and in March at Chicago, Ill.).

During the year the appointment of a new Subcommittee XVIII on Aniline Point was authorized and this subcommittee is now being organized.

The following revisions of American standards and American tentative standards have been approved by the American Standards Association, on the recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants, to bring these standards into conformity with the revisions in the A.S.T.M. standards approved during the past year:

American Tentative Standards

	A.S.T.M. Designation	A.S.A. Number
Method of Test for Knock Characteristics of Motor Fuels	D 357 – 36 T	Z11.37–1936
Definitions of Terms Relating to Petroleum............	D 288 – 36 T	Z11.28–1936

American Standards

	A.S.T.M. Designation	A.S.A. Number
Abridged Volume Correction Table for Petroleum Oils...	D 206 – 36	Z11.1 –1936
Methods of Test for:		
Burning Quality of Kerosine Oils..................	D 187 – 36	Z11.17–1936
Burning Quality of Long-Time Burning Oil for Railway Use.............................	D 219 – 36	Z11.19–1936
Carbon Residue of Petroleum Products (Conradson Carbon Residue)........................	D 189 – 36	Z11.25–1936
Distillation of Crude Petroleum..................	D 285 – 36	Z11.32–1936
Steam Emulsion of Lubricating Oils................	D 157 – 36	Z11.15–1936
Flash Point by Means of the Pensky-Martens Closed Tester..............................	D 93 – 36	Z11.7 –1936
Flash Point by Means of the Tag Closed Tester.......	D 56 – 36	Z11.24–1936
Gravity of Petroleum and Petroleum Products by Means of the Hydrometer........................	D 287 – 36	Z11.31–1936
Saponification Number........................	D 94 – 36	Z11.20–1936
Viscosity by Means of the Saybolt Viscosimeter.......	D 88 – 36	Z11.2 –1936

Also the American Tentative Standard Method of Test for Gum Content of Gasoline (A.S.T.M. Designation: D 381 – 36) (A.S.A. No. Z11.36–1936) was revised and approved as American standard.

Acting on the recommendation of Committee D-2, Committee E-10 on Standards[1] on August 26, 1936, accepted a revision of the Tentative Definitions of Terms Relating to Petroleum (D 288 – 35 T), and on March 30,

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-2 reported results of the letter ballot vote as follows: On Definitions D 288, of a total membership of 87, 62 members returned their ballots, of whom 60 voted affirmatively, 0 negatively, and 2 marked their ballots "not voting;" on Specifications D 439, of a total membership of 84, 67 members returned their ballots, of whom 56 voted affirmatively, 5 negatively, and 6 marked their ballots "not voting."

1937, accepted for publication the Tentative Specifications for Gasoline (D 439 – 37 T).[1] The definitions in their revised form appear in the 1936 Proceedings.[2]

The voting membership of Committee D-2 now consists of 39 producer, 31 consumer, and 14 general interest members.

Activities of Subcommittees and Technical Committees

The reports of the following subcommittees are appended hereto: Subcommittee XI on Turbine Oils and Subcommittee XXII on Natural Gasoline.

Subcommittee IV on Grease (Ralph R. Matthews, chairman) has prepared a description of a Proposed Method of Test for Dropping Point of Lubricating Greases which is published as information in Appendix I; also a minor revision of the Standard Methods of Analysis of Grease (D 128 – 27) given in detail later in this report.

Subcommittee V on Viscosity (J. C. Geniesse, chairman) has developed new proposed Tentative Methods of Test for Kinematic Viscosity and a Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity, as appended hereto;[3] also a revision of the Tentative Standard Viscosity-Temperature Chart for Liquid Petroleum Products (D 341 – 32 T). The methods of test for kinematic viscosity represent a consolidation and revision of the methods published as information in the 1936 report of Committee D-2.[4] In connection with the new method for viscosity conversion, C. E. Headington and J. C. Geniesse have prepared a brief paper on "Method for Converting Kinematic Viscosity to Saybolt Universal Viscosity" which appears in Appendix III. The revised viscosity-temperature chart covers a viscosity range of from 33 to 100,000,000 Saybolt Universal seconds and a temperature range of –30 to +450 F. The spaces between the lines at low viscosities have been slightly altered in order to eliminate the slight curvature frequently obtained with the present chart. The lines are also set at a small angle from the horizontal in order to take into account the fact that the Saybolt efflux time, for a given kinematic viscosity, varies with temperature. Also three other charts are to be included, an abridged Saybolt Universal chart covering a viscosity range of 33 to 100,000 seconds and a temperature range of –10 to 300 F., and two kinematic charts, both of the same temperature range as the larger Saybolt chart and with viscosity ranges of 2 to 20,000,000 and 0.4 to 100 centistokes, respectively.

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[1] See p. 872.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 857 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 669.

[3] See pp. 900 and 911.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 394, 399 (1936).

Subcommittee XVII on Plant Spray Oils (J. B. Terry, chairman) is recommending a new proposed Tentative Method of Test for Distillation of Plant Spray Oils, appended hereto,[1] based upon the gas oil distillation method, and also has recommended that a Proposed Method of Test for Unsulfonated Residue of Plant Spray Oils, in Appendix II, be published as information.

Technical Committee A on Gasoline (C. B. Veal, chairman) has prepared Tentative Specifications for Gasoline (D 439 - 37 T)[2] for use in contract purchases. Sections of this technical committee are actively engaged in the development of methods of test for acid heat, gum stability, and tetraethyl lead in gasoline.

Technical Committee B on Motor Oils (H. C. Mougey, chairman) arranged a Symposium on Lubricants[3] which was held at the regional meeting of the Society in Chicago on March 3, 1937.

Technical Committee C on Fuel Oils (*Including Diesel Fuel*) (M. J. Reed, chairman) has under consideration a revision of the Tentative Specifications for Fuel Oils (D 396 - 34 T). The committee has also revised the Diesel-Fuel-Oil Classification to include a provision that oils Nos. 1D, 3D, 4D and 5D shall be free from alkali and mineral acid. The revised classification is published as information in Appendix IV.

RECOMMENDATIONS AFFECTING STANDARDS

I. Proposed Tentative Standards:

The committee recommends that the following four methods of test be accepted for publication as tentative, as appended hereto:[4]

Tentative Methods of:

Test for Kinematic Viscosity, as proposed by Subcommittee V;

Test for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity, as proposed by Subcommittee V;

Test for Distillation of Plant Spray Oils, as proposed by Subcommittee XVII;

Test for Vapor Pressure of Petroleum Products (Reid Method), proposed by Subcommittee XXII as a consolidation and revision of the present Tentative Methods of Test for Vapor Pressure of Natural Gasoline (Reid Method) (D 323 - 32 T) and of Motor and Aviation Gasoline (Reid Method) (D 417 - 35 T), which are accordingly recommended to be withdrawn.

II. Proposed Revisions of Tentative Standards:

The committee recommends revisions of two tentative standards, as follows:

Tentative Method of Test for Knock Characteristics of Motor Fuels (D 357 - 36 T).[5]—The following revision is recommended by Technical

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[1] See p. 875.

[2] See p. 872.

[3] Symposium on Lubricants, Chicago Regional Meeting, Am. Soc. Testing Mats. (1937). (Available as separate publication.)

[4] See pp. 900, 911, 875 and 888.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 848 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 620.

Committee A on Gasoline (C. B. Veal, chairman) to bring the test in line with present practice regarding the use of secondary reference fuels:

Section 5.—Letter the present paragraph as Paragraph (*a*) and add as Paragraph (*b*) the present Note 1, modified to read as follows:

(*b*) *Secondary Reference Fuels.*—To insure a greater degree of reproducibility in testing, it is preferable to use secondary[a] reference fuels which have been properly calibrated against the primary reference fuels on a large number of engines. Such secondary reference fuels may be straight-run or other stable gasolines suitable for the purpose. One of the reference fuels should be of low knock rating and the other of high knock rating. These secondary reference fuels shall be calibrated against normal heptane and *iso*octane. The calibration shall be expressed in terms of the octane-number scale, shall be based on results by this method from at least ten laboratories, and shall be certified by the National Bureau of Standards (Note 2).

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[a] Such secondary reference fuels are commercially available through the Standard Oil Company of New Jersey with calibration curves as determined in accordance with this method, giving octane numbers for these fuels blended with each other. They may be ordered from the Standard Oil Development Co., Elizabeth, N. J.

Add a new note as Note 2 to read as follows, renumbering the present Note 2 as Note 1:

NOTE 2.—For the present this certification is based on tests made by members of the C.F.R. Motor Fuels Committee on samples from each batch of secondary reference fuels, and on blends of the high and low secondary fuels against blends of *iso*octane and normal heptane. A certificate is then issued to the producers authorizing them to guarantee to the purchasers that the material shipped is a part of a batch so tested, and to quote the results of the tests.

Tentative Standard Viscosity-Temperature Chart for Liquid Petroleum Products (D 341 – 32 T).[1]—Four viscosity-temperature charts[2] prepared by Subcommittee V on Viscosity (J. C. Geniesse, chairman) are recommended to replace the existing Tentative Standard D 341 – 32 T.

III. Proposed Revision of Standards:

The committee recommends the revision of three standard methods as indicated below, to become effective immediately. Accordingly, the necessary nine-tenths vote at the annual meeting is requested in order that these revisions may be referred to letter ballot of the Society.

Standard Methods of Analysis of Grease (D 128 – 27).[3]—The following revision is recommended by Subcommittee IV on Grease (Ralph R. Matthews, chairman) to improve the method for determination of free alkali or free acid:

Section 5.—Change to read as follows by the omission of the words in brackets:

5. The alcohol shall be prepared from commercial 95 per cent grain or denatured grain alcohol by distilling from sodium hydroxide, [diluting to 50 per cent (or 70 per cent) by volume with distilled water] and neutralizing exactly with sodium or potassium hydroxide and phenolphthalein.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 772 (1932); also 1936 Book of A.S.T.M. Tentative Standards, p. 666.

[2] See p. 914.

[3] 1936 Book of A.S.T.M. Standards, Part II, p. 926.

Section 23.—Change the first sentence to read as follows by the addition of the italicized words and figure and the omission of the figure in brackets:

> From 10 to 30 g. of grease shall be weighed in a small beaker, dissolved as completely as possible in 75 ml. of petroleum ether by stirring with a spatula, the mixture washed into a 250-ml. Erlenmeyer flask with a small amount of petroleum ether, and the beaker rinsed with 50 ml. of [50] *95* per cent alcohol (*See Section 5*), the alcohol being poured into the flask, after which a few drops of phenolphthalein solution shall be added and the [whole] *contents* shaken vigorously.

Standard Method of Test for Melting Point of Paraffin Wax (D 87-22).[1]—The following revision is recommended by Subcommittee III on Paraffin Wax (J. B. Rather, chairman) to change the range of the thermometer, which is specified in the Standard Method of Test for Melting Point of Petrolatum (D 127), and thus provide for the testing of petrolatums of melting points higher than 160 F.:

Section 6.—Change the requirements for the ranges of the A.S.T.M. paraffin wax melting point thermometers to read as follows by the addition of the italicized figures and the omission of those in brackets:

> Range and Subdivision: [27] *38* to [71] *82* C. in 0.1 C. or [80] *100* to [160] *180* F. in 0.2 F.

Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (D 287 - 36).[2]—The following revision is recommended by Subcommittee II on Specific Gravity (H. T. Bennett, chairman) in order to improve the description of the hydrometer to permit the use of other than paper scales:

Section 3.—Change the second sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> It shall be of glass of the conventional constant-mass and variable-displacement type, with a [paper hand-written] scale *of suitable material* [printed in] *clearly marked with* black ink.

In the eighth and ninth sentences omit the word "paper" in the description of the hydrometer scale; also in the eighth sentence, substitute the word "marked" for "printed."

Section 4 (b).—Change this section to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> (*b*) Thermometer for Thermo-hydrometer.—The thermometer incorporated in the thermo-hydrometer type of instrument shall [be of the paper scale type (scale to be placed in either the bulb or stem of the hydrometer) having] *have* a range of from 20 to 130 F. or 60 to 220 F. depending upon the range of the hydrometer. The [paper] temperature scale shall be graduated in 2 F. divisions accurate to ±1 F. (Note) and, when located in the stem of the hydrometer, shall be [printed] *marked* in red ink to avoid any possibility of its being confused with the black gravity scale.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 938.

[2] *Ibid.*, p. 918.

IV. Tentative Revision of Standard:

Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (D 88 – 36).[1]—The following revision of this method proposed by Subcommittee V on Viscosity (J. C. Geniesse, chairman) in order to take care of certain difficulties encountered with cut-back asphalts is recommended for publication as tentative:

Section 4 (c).—Change the note following this section to read as follows by the addition of the italicized sentence:

NOTE.—The plunger commonly supplied with the viscosimeter shall never be used on instruments maintained as standards. ***In case the flash point is less than 50 F. above the temperature of test, the wetting operation should be omitted in order to avoid an increase in viscosity due to evaporation of a portion of the light fraction.***

Section 4 (d).—After the first sentence add a new sentence to read as follows:

In case the flash point is less than 50 F. (10 C.) above the temperature of test, the oil shall be heated to room temperature only.

V. Tentative Standards Continued as Tentative:

Seven methods of test have been tentative for a period of three years or longer without revision, as follows:

Tentative Methods of:

Test for Color of Lubricating Oils by Means of A.S.T.M. Union Colorimeter (D 155 – 34 T),
Test for Color of Petrolatum by Means of A.S.T.M. Union Colorimeter (D 218 – 34 T),
Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (D 156 – 34 T),
Test for Neutralization Number of Petroleum Products and Lubricants (D 188 – 27 T),
Test for Expressible Oil and Moisture in Paraffin Waxes (D 308 – 29 T),
Test for Penetration of Greases and Petrolatum (D 217 – 33 T), and
Test for Sulfur in Petroleum Oils by Lamp Method (D 90 – 34 T).

The fifth of these methods is a highly empirical one and is used only infrequently. The other methods may be revised before being advanced to standard as a result of work now under way. It is accordingly recommended that these methods be continued as tentative.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 84 members; with the results shown in Table I.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 982.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. PROPOSED TENTATIVE STANDARDS			
Test for Kinematic Viscosity	64	0	4
Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity	64	0	4
Test for Distillation of Plant Spray Oils	53	0	15
Test for Vapor Pressure of Petroleum Products (Reid Method) to replace Methods D 323 - 32 T and D 417 - 35 T	63	0	5
II. PROPOSED REVISION OF TENTATIVE STANDARDS			
Test for Knock Characteristics of Motor Fuels (D 357 - 36 T)	53	0	15
Viscosity-Temperature Chart for Liquid Petroleum Products (D 341 - 32 T)	61	2	5
III. PROPOSED REVISION OF STANDARDS			
Analysis of Grease (D 128 - 27), immediate adoption	60	1	7
Test for Melting Point of Paraffin Wax (D 87 - 22), immediate adoption	60	1	7
Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (D 287 - 36), immediate adoption	70	0	0
IV. TENTATIVE REVISION OF STANDARD			
Test for Viscosity by Means of the Saybolt Viscosimeter (D 88 - 36)	68	2	0

This report has been submitted to letter ballot of the committee which consists of 84 members; 68 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

T. A. BOYD,
Chairman.

R. P. ANDERSON,
Secretary.

REPORT OF SUBCOMMITTEE XI ON TURBINE OILS

Subcommittee XI on Turbine Oils has attempted to ascertain what A.S.T.M. standard methods of test are applied by turbine operators to new oils and what other methods, not standard, are in such general use as to warrant collection of details and of results to be expected for the benefit of others.

To obtain the information needed, the cooperative assistance of the Chemist's Subcommittee of the Edison Electric Institute was enlisted. This committee sent to a representative list of 34 operators a questionnaire indicating the four chief objectives of the cooperative investigation as follows:

1. To ascertain what A.S.T.M. methods of test under the jurisdiction of Committee D-2 are used by turbine operators in testing and evaluating their steam-turbine lubricants.
2. To ascertain what other methods are used by operators to evaluate their lubricants for steam turbines.
3. To obtain full details of methods not standardized by the A.S.T.M. in order that others may apply them.
4. To obtain statements of the reasons why other than A.S.T.M. standard methods are preferred, the results yielded by such methods, and statements of their value and their bearing on the usefulness of the lubricant.

The responses to question 1 were not always specific and definite, but may be summarized as follows: Out of 34 companies contacted, 25 replies were received. The A.S.T.M. methods used and the number of operators using them are as follows:

A.S.T.M. Method	Number of Operators Using Test for Turbine Lubricants
Specific gravity	16
Viscosity	21
Flash point	15
Fire point	15
Pour point	6
Neutralization number	16
Color	10
Steam emulsion	15

The answers to question 2 showed that there were no other methods in such general use as to warrant consideration by the committee at present, although there are other methods used which may in the future develop to the point of requiring such consideration.

In view of the answers to question 2, no data on questions 3 and 4 are available until methods used by individuals have developed to the point where they warrant consideration.

The replies to the questionnaire show a general use of A.S.T.M. methods. They also indicate an interest in the development of a type of test which would permit selection of a turbine lubricant with reasonable assurance of satisfactory performance. The development of such a test would be a matter of research.

Respectfully submitted on behalf of the subcommittee,

J. A. CAPP,
Chairman.

REPORT OF SUBCOMMITTEE XXII ON NATURAL GASOLINE

When the Tentative Method of Test for Vapor Pressure of Motor and Aviation Gasoline (Reid Method) (D 417 – 35 T),[1] was issued by the Society in 1935, a note was included in Section 1 on Scope reading as follows:

> NOTE.—Present indications are that reliable results can be obtained by this method on gasolines of 28-lb. vapor pressure, or less. Probable exceptions are gasolines of somewhat lower vapor pressure containing appreciable quantities of dissolved gases.

This note raises three questions: (*1*) Is 28 lb. the proper upper limit of application of Method D 417; (*2*) is Method D 417 practical for use in natural-gasoline plant laboratories; and (*3*) what vapor-pressure method should be prescribed for natural gasolines above the upper limit of application of Method D 417?

It was evident that the Tentative Method of Test for Vapor Pressure of Natural Gasoline (Reid Method) (D 323 – 32 T),[2] revised to include the refinements incorporated in Method D 417, but omitting the air-saturation step, would be the answer to the third question.

To determine the answer to question 1, two series of experiments have been conducted. The first of these was conducted by the Technical Committee of the California Natural Gasoline Association in the spring of 1935. In these tests, vapor-pressure determinations by Methods D 417 and D 323, essentially as described in the revised methods appended hereto,[3] were compared. Motor fuels and rectified and unrectified natural gasolines were studied. One hundred vapor-pressure determinations were made on fifteen stocks ranging in nominal vapor pressure from 6 to 50 lb. The results of these tests are summarized graphically in Fig. 1. As a result of these experiments, the committee recommended the application of the air-saturation method (Method D 417 as revised) to all natural gasolines having Reid values below 28 lb. and the use of the air-free method (Method D 323 as revised) to all natural gasolines having Reid values above 26 lb.

The second series of tests was conducted by the Technical Committee of the Natural Gasoline Association of America in the fall of 1935. In these experiments all the samples were initially air-saturated, the object being to emphasize the effect of evaporation in Method D 417. Both well-rectified and poorly rectified gasolines were studied. The results of these tests are summarized graphically in Fig. 2.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 903 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 648.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 765 (1932); also 1936 Book of A.S.T.M. Tentative Standards, p. 659.

[3] See p. 888.

Comparing these two sets of carefully conducted experiments, one minor and one major point should be mentioned. The minor point is that Method D 417 gave higher results relative to Method D 323 in the C.N.G.A. experiments (first series) than in the N.G.A.A. experiments (second series). This is to be accounted for by the fact that in the N.G.A.A. experiments the materials tested were deliberately saturated with air prior to sampling, whereas this was not the procedure with the C.N.G.A. experiments.

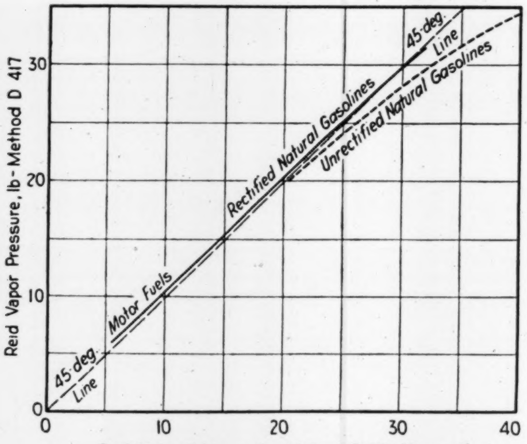

FIG. 1.—Comparative Reid Vapor Pressure Tests: Method D 417 *versus* Method D 323 (Revised).

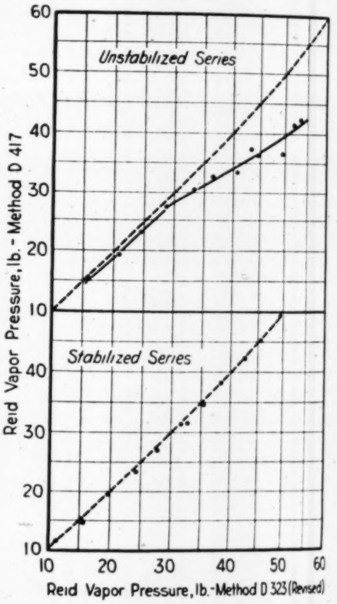

FIG. 2.—Comparative Reid Vapor Pressure Tests: Method D 417 *versus* Method D 323 (Revised).

The major point is that there is substantial agreement in regard to the upper limit of applicability of Method D 417. With well-rectified materials this upper limit could be considerably more than 26 lb. With poorly fractionated material the limit could not safely be extended above 26 lb. These experiments therefore constitute the basis for the recommendation incorporated in the revised methods appended hereto.[1]

The second question created by the adoption of Method D 417 (practicability of its use in natural-gasoline operations) has two phases: (*a*) convenience and cost merely as a testing method; and (*b*) economic effects. In regard to the former, it was realized that the air-saturation requirement of Method D 417 introduces a considerable item of added expense in plant testing not heretofore considered necessary. The extra steps of intermediate sampling and air-saturation would require many natural-gasoline plant laboratories to invest in new testing equipment and to increase testing personnel to some extent. However, it was further realized that these factors were not of themselves sufficient justification for two A.S.T.M. methods in the same field of testing. Consequently, it was decided by the N.G.A.A. to submit the two testing methods to the acid test of commercial application to determine what the economic effect would be.

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[1] See p. 888.

TABLE I.—DIFFERENCES IN REID VAPOR PRESSURE RESULTS IN TESTS DETERMINED BY METHOD (D 417 – 35 T AND METHOD D 323 (REVISED)) ON TANK-CAR SHIPMENTS OF NATURAL GASOLINE (POINT OF ORIGIN ONLY).

	Nominal Reid Vapor Pressure, lb.				
	12	14	18	22	26
Number of samples tested	33	184	177	99	230
Average Reid vapor pressure, lb. Method D 417	12.71	13.80	17.81	21.52	25.63
Average Reid vapor pressure, lb. Method D 323	12.35	13.59	17.55	21.28	25.67
Average difference in vapor pressure, lb.	0.36	0.21	0.26	0.24	0.04
Number of Samples of Which Result by Method D 417 were:					
1.0 to 2.0 lb. lower	......	......	1	......	4
Percentage of total	......	......	0.5	......	1
0.5 to 0.9 lb. lower	......	......	1	......	10
Percentage of total	......	......	0.5	......	5
0.1 to 0.4 lb. lower	......	12	8	9	82
Percentage of total	......	7	4	9	35
equal to those of Method D 323	2	15	8	8	44
Percentage of total	6	8	4	8	17
0.1 to 0.5 lb. higher	31	154	149	80	87
Percentage of total	94	84	85	81	37
0.6 to 1.0 lb. higher	......	3	9	2	10
Percentage of total	......	0.1	4	2	5
1.0 to 2.0 lb. higher	......	......	1	......	......
Percentage of total	......	......	0.5	......	......

TABLE II.—COMPARISONS OF REID VAPOR PRESSURE DETERMINATIONS (RESULTS BY METHOD D 417 – 35 T MINUS D 323 (REVISED)) AT ORIGIN AND DESTINATION.

Number of Tank Cars Tested	Average Reid Vapor Pressure, lb.					
	Tested at Place of Origin			Tested at Place of Destination		
	Method D 417	Method D 323	Difference	Method D 417	Method D 323	Difference
24	13.94	13.76	+0.18	13.43	13.19	+0.24
47	17.85	17.53	+0.33	17.62	17.51	+0.11
22	21.72	21.35	+0.37	21.01	20.83	+0.18

During the six months, March to August, 1936, N.G.A.A. member companies ran tests on a total of 814 tank-car shipments of natural gasoline. These tests involved a comparison of vapor-pressure determinations obtained by Methods D 417 and D 323, essentially as revised herewith.[1] Of the 814 car loads of gasoline so tested, 93 were tested at both point of origin and at destination. The data so accumulated were summarized by the N.G.A.A. Technical Committee and are presented in abbreviated form in Tables I and II.

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[1] See p. 888.

These tests demonstrate that in actual application the substitution of Method D 417 for Method D 323 for natural gasolines up to and including 26-lb. vapor pressure, will impose a slight economic hardship on the natural-gasoline manufacturer. The summer season for running these tests was deliberately chosen because on theoretical grounds the adverse difference between the two methods should be greater in warm weather. In view of the fact that Method D 417 provides the refiner with a more accurate and convenient method in his normal operations than does Method D 323, the N.G.A.A. here again decided that the economic handicap thus demonstrated does not justify two A.S.T.M. methods of test covering the same field of testing.

While the experimental data herewith reported have the most critical and significant bearing on the subject, a vast amount of additional experimental work concerned with these testing methods has been done and the conclusions derived from it are in excellent agreement with the data submitted.

Subcommittee XXII therefore recommends the consolidation of the two vapor-pressure methods into a single method as appended hereto.[1]

Respectfully submitted on behalf of the subcommittee,

G. G. OBERFELL,
Chairman.

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[1] See p. 888.

APPENDIX I

PROPOSED METHOD OF TEST
FOR
DROPPING POINT OF LUBRICATING GREASES[1]

This is a **proposed method** and is published as information only. Comments are solicited and should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

1. *Scope.*—This method is intended for use in determining the dropping point of lubricating greases.

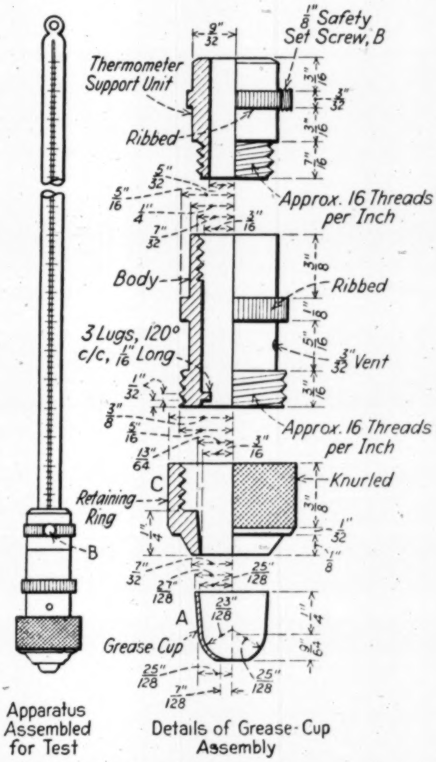
1/8" Safety Set Screw, B
9/32"
Thermometer Support Unit
3/16"
3/32"
Ribbed
3/16"
7/16"
5/32"
5/16"
1/4"
3/16"
7/32"
Approx. 16 Threads per Inch
Body
3/8"
Ribbed
1/8"
3 Lugs, 120° c/c, 1/16" Long
5/16"
3/32" Vent
1/32"
3/16"
3/8"
Approx. 16 Threads per Inch
5/16"
3/16"
13/64"
C
Knurled
Retaining Ring
3/8"
1/4"
1/32"
B
1/8"
7/32"
25/128"
27/128"
A
23/128"
1/4"
Grease Cup
3/64"
25/128"
25/128"
7/128"
Apparatus Assembled for Test
Details of Grease-Cup Assembly

FIG. 1.—Apparatus for Testing Dropping Point of Greases.

When assembled for test, the lower end of the bulb shall be placed exactly 1⅛ in. above the bottom of the grease cup.

NOTE.—The dropping point as determined by this method gives information as to when softening begins under the conditions of the test, but should not be considered as having any bearing upon performance in service.

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[1] Under the standardization procedure of the Society, this proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

2. *Apparatus.*—(*a*) *Thermometer.*—The thermometer shall be graduated in Fahrenheit degrees, the range being 20 to 580 F., and shall conform to the Standard Specifications for A.S.T.M. Partial-Immersion Thermometer for General Use (A.S.T.M. Designation: D 183) of the American Society for Testing Materials.[1]

(*b*) *Grease-Cup Assembly.*—The grease-cup assembly shall conform to the requirements shown in Fig. 1.

(*c*) *Accessories.*—Accessories required consist of a test tube approximately 4 cm. in diameter by 18 cm. in height, a 600-ml. beaker, ring stand, clamp for test tube, support for beaker, gas burner, suitable oil[2] for oil bath, etc.

3. *Procedure.*—(*a*) The grease-cup assembly shall be placed in position on the thermometer at a height so that the tip of the thermometer bulb will be $\frac{1}{8}$ in. above the bottom of the grease cup, *A*. The set screw, *B*, shall then be tightened to hold the assembly in position on the thermometer.

(*b*) The grease cup shall then be removed and filled with grease by pressing the larger end into the grease to be tested until the cup is filled, taking care to avoid inclusion of air. Working the grease shall be avoided as far as possible. Excess grease shall be removed with a spatula or knife.

(*c*) The full grease cup shall then be placed on the retaining ring, *C*, and the ring screwed in position to bring the thermometer bulb to the original position with respect to the cup outlet. Grease forced out in this operation shall be carefully removed.

(*d*) The thermometer and assembly shall then be placed in the test tube with the bottom of the cup 1 in. from the bottom of the test tube and the test tube suspended in the oil bath submerged to one-half its height.

(*e*) The oil bath shall be heated so as to cause the temperature indicated by the thermometer to rise at a rate of 8 to 10 F. per min. until a temperature approximately 30 F. below the expected dropping point of the grease is reached. At this point, the rate of heating shall be changed to 2 to 3 F. per min. As the temperature increases, grease will gradually protrude through the orifice of the grease cup. When a drop of grease falls the temperature shall be noted and recorded as the dropping point.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1405.

[2] A suitable oil is one that does not vaporize appreciably at the temperature of the test and does not darken rapidly with repeated heating. Castor oil is satisfactory.

APPENDIX II

PROPOSED METHOD OF TEST
FOR
UNSULFONATED RESIDUE OF PLANT SPRAY OILS[1]

This is a **proposed method** and is published as information only. Comments are solicited and should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Scope

1. This method is intended for use in determining the amount of unsulfonated residue of plant spray oils.

Special Solutions Required

2. (*a*) *Sulfuric Acid (37 N).*—Prepare a 37 *N* sulfuric acid solution by blending reagent-grade fuming and concentrated sulfuric acids to 98.61 per cent ± 0.2 per cent H_2SO_4 at a temperature of 59 F. (15 C.) to be determined by accurate titration.

(*b*) *Sulfuric Acid (sp. gr. 1.84).*—Concentrated H_2SO_4, approximately 1.84 sp. gr.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Sample Bottles.*—Ordinary 4-oz. glass bottles for oil samples.

(*b*) *Pipette.*—One 5-ml. pipette, precision grade, calibrated in the laboratory to deliver 5 ml. ± 0.005 ml. of oil at 77 F.

(*c*) *Burette.*—One 50-ml. burette, graduated in 0.1-ml. divisions.

(*d*) *Test Bottles.*—The test bottles shall be made of good quality glass and the preferred form shall conform to the dimensions given in Fig. 1.[2] The capacity to base of neck shall be 45 ml. ± 1.0 ml. As an alternative the standard 6.5-in., 50 per cent cream bottle may be used provided the same accuracy is maintained. The graduated portion of the bottle shall contain 5 ml. ± 0.02 ml. at a temperature of 77 F. (25 C.) and shall be graduated from 0 to 50 per cent in 0.5 per cent divisions, the first and each succeeding 5 per cent line to extend at least three-fourths of the distance around the neck and to be numbered from the bottom. Within the range from 0 to 50 per cent, the maximum error in volume shall not be greater than 0.02 ml. The graduation marks shall be clear and fine, not more than 0.3 mm. in width. The bottom of the bottles shall have a ground area of at least 2 sq. cm. for numbering.

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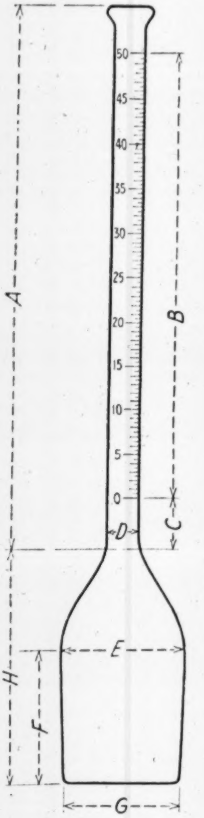
[1] Under the standardization procedure of the Society, this proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Commonly known as "9-in., 9-g., 50 per cent x ½ cream bottles."

(*e*) *Water Baths.*—Two water baths will be required, as follows:

(*1*) A water bath maintained at 77 F. ± 0.5 F. (25 C. ± 0.3 C.) and of such depth that regular 4-oz. bottles containing samples for test may be immersed, the oil level being well below the surface of the water.



A = 16 cm. ± 0.5 cm.
B = 13 cm. ± 1.0 cm.
C = not less than 0.5 cm.
D = 0.70 cm. ± 0.05 cm., inside diameter.
E = not more than 3.75 cm.
F = approximately 4 cm.
G = not more than 3.75 cm.
H = approximately 6.5 cm.
A + H = 22.5 cm. ± 0.5 cm.

FIG. 1.—Test Bottle.

(*2*) A water bath maintained at 211 to 212 F. (99 to 100 C.), of sufficient depth to permit complete immersion of the body of the test bottle and equipped with clamps to hold the bottle rigidly in a vertical position.

(*f*) *Centrifuge.*—A centrifuge, capable of whirling at the required speed two test bottles filled with acid. The preferred form of centrifuge shall have a diameter of swing (at the base of whirling bottles) sufficient to accommodate the test bottles without danger of interference when placed in opposite carriers and a speed of 1000 r.p.m. The centrifuge shall be of good design and rugged construction, so that it may be operated without danger. The bottle carrier shall be so designed that the test bottles may be cushioned with rubber or other suitable material. A strong metal shield, approximately 2 ft. in diameter, shall be placed around the centrifuge to eliminate danger if breakage occurs.

(*g*) *Mechanical Shaker.*—A preferred form of mechanical shaker constructed in accordance with the requirements shown in Fig. 2. This machine shall shake the test bottles at a rate of 200 to 220 complete shakes per minute, this rate being determined by attaching a counter to engage on the moving platform and maintained by installing a suitable rheostat to control fluctuations in line voltage.

Procedure

4. (*a*) A small portion (15 or 20 ml.) of the sample under test shall be placed in a clean, dry, 4-oz. bottle, which shall then be immersed in the water bath at 77 F. (25 C.) until the sample has attained bath temperature Exactly 5 ml. of the sample shall then be drawn into the clean, dried, especially calibrated pipette and delivered into the clean, dried test bottle.

NOTE.—As an alternative procedure, the 5-ml. sample may be obtained by weighing. The weight of the sample shall agree within ±0.005 g. of the weight of 5 ml. of the oil at 77 F. (25 C.), as calculated from its specific gravity.

(*b*) Five milliliters of the 37 *N* sulfuric acid shall then be slowly added to the test bottle from the burette in such a way that the acid washes down any oil remaining in the neck of the bottle. The test bottle shall then be shaken for 2 or 3 sec., grasping the neck between the thumb and index finger and swinging the bottle through an arc of approximately 20 deg., such that the bottom of the bottle passes through a distance of $2\frac{1}{2}$ to $3\frac{1}{2}$ in. The speed of shaking shall be maintained at from 240 to 300 complete swings per minute. The temperature of this acid-oil mixture shall not be allowed to exceed 212 F. (100 C.), as indicated by the bottle becoming uncomfortable to the touch, or the contents foaming excessively. The bottle may be cooled in ice water if necessary.

(*c*) The procedure outlined in Paragraph (*b*) shall be repeated immediately until a total of 20.0 ml. of 37 *N* sulfuric acid has been added, the test bottle being placed finally in the water bath at 212 F. (100 C.). These several additions of acid should be made without delay.

(*d*) After the test bottle has been in the bath for 10 min., it shall be removed, placed immediately on the mechanical shaker and agitated at a rate of 200 to 220 shakes per minute for 10 sec. The bottle shall then be replaced immediately in the water bath at 212 F. (100 C.). If a sample boils or foams over at this stage, it must be discarded and the test repeated.

(*e*) The procedure outlined in Paragraph (*d*) shall be repeated for a period of 1 hr. from the time the test bottle was first immersed in the water bath at 212 F. (100 C.).

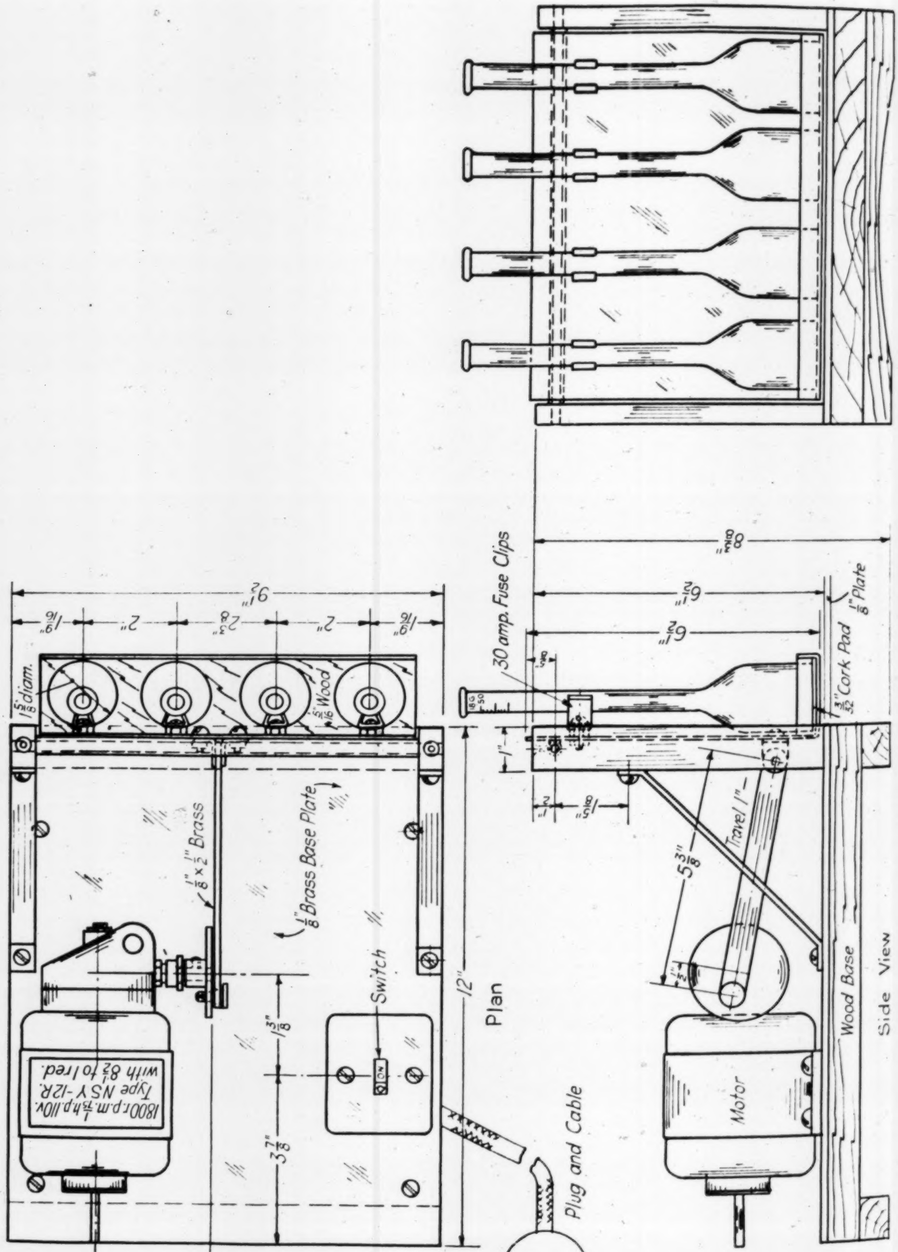

9 1/2"
1 9/16"
2"
2 3/8"
2"
1 9/16"
1 5/8" diam.
5/16" Wood
1/8" × 1/2" Brass
1/8" Brass Base Plate
Switch
ON
1 3/8"
3 7/8"
12"
Plan
5 1/8"
1800 r.p.m. 1/15 h.p. 110 v.
Type NSY-12R.
with 8 1/2 to 1 red.
Plug and Cable
30 amp. Fuse Clips
8 3/4"
6 1/2"
6 1/2"
1/8" Plate
3/32" Cork Pad
1"
1/2"
1 5/8"
5 3/8"
Travel 1"
1 1/2"
Motor
Wood Base
Side View

FIG. 2.—Mechanical Shaker.

(*f*) The test bottle shall then be removed from the water bath, and sufficient sulfuric acid (sp. gr. 1.84) added to raise the liquid level in the neck of the bottle to the 50 per cent mark, that is, the top graduation. The bottle and contents shall then be placed in the centrifuge and whirled as prescribed in Section 3 (*f*) at a speed of approximately 1000 r.p.m. for 10 min. The bottle shall then be removed from the centrifuge and placed in the water bath at 77 F. (25 C.) so that the entire contents of the bottle are immersed below the surface of the water. After 10 min., the bottle shall be removed from the bath and the volume of oil read with the aid of a reading glass, the reading being estimated to within 0.01 ml. The centrifuging process shall be repeated until the volume of oil is constant.

(*g*) After a constant oil volume has been obtained by centrifuging (Paragraph (*f*)), the reading of the bottom of the oil level shall be subtracted from the reading of the top and the difference multiplied by two. This value shall be reported to the nearest 0.1 ml. and is the percentage of unsulfonated residue. Due attention shall be paid to calibration corrections.

Precautions

5. (*a*) It is extremely important that all glassware used in the test shall have been previously thoroughly cleaned and dried. Acetone has proved satisfactory for this purpose.

(*b*) If at any time during the test even the smallest quantity of oil or acid-oil mixture is lost, the sample shall be discarded and the test repeated.

(*c*) The rate of whirling may be decreased to avoid breakage of the test bottles. In all cases, however, the centrifuging shall be continued until a constant reading of the volume of oil is obtained. Ordinarily, centrifuging at 1000 r.p.m. for 10 min. is sufficient, but occasionally oils are encountered which do not separate readily and completely from the acid sludge.

Reproducibility of Results

6. For the most satisfactory results, determinations should be made in duplicate, and should agree within 0.5 per cent unsulfonated residue based upon the original sample. This accuracy should be expected only when proper care and strict attention to details are observed. Determinations by different operators should agree within 1.0 per cent.

APPENDIX III

METHOD FOR CONVERTING KINEMATIC VISCOSITY TO SAYBOLT UNIVERSAL VISCOSITY

By C. E. Headington[1] and J. C. Geniesse[1]

In 1936 Committee D-2 published, as information, two methods for measuring kinematic viscosity.[2] These were submitted to the industry for trial in response to a demand for a more accurate method of measuring viscosity than the Saybolt instrument provides. This year the Society is publishing these kinematic methods as a tentative standard.[3]

This trend in the petroleum industry toward the measurement of viscosity in kinematic units is developing a real need for a reliable method for converting these viscosity units to Saybolt Universal seconds since it is apparent that the industry will continue to specify Saybolt seconds for some time to come.

The problem was referred to Subcommittee V on Viscosity. A sub-subcommittee consisting of members who had had considerable experience with various methods of measuring viscosity was appointed to undertake the experimental work. Four of these members,[4] whose kinematic equipment was among the best available in the industry, were selected for kinematic viscosity measurements. Four other members,[5] whose Saybolt tubes were practically zero-correction instruments and therefore represented as closely as possible the proposed A.P.I. Saybolt Master Tube, were selected for Saybolt viscosity measurements.

Previous Conversions:

At the time the work was started, five different conversions had been developed from experimental results, no two of which were entirely in agreement. An examination of these five independently developed conversions,[6] however, disclosed the fact that there was excellent agreement between four of them from 150 to 1000 Saybolt Universal sec. (32 to 216 centistokes). At no point between 36 and 4000 Saybolt sec. (3 to 864 centistokes) did they deviate by more than 2 per cent from the average of all five. This is shown in Fig. 1 where for given kinematic viscosities the deviation of the Saybolt equivalents from the average for each of the five conversions is plotted.

In view of this agreement it seemed advisable to use the average of these five conversions as the basis for a new one. With this procedure a table of average Saybolt equivalents for given kinematic viscosities was available at the outset; consequently relatively few experimental samples were required to reveal the deviation of these average Saybolt equivalents, or basic averages, from the actual experimental results over the whole Saybolt range.

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[1] Research Chemist, The Atlantic Refining Co., Philadelphia, Pa.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 394 and 399 (1936).

[3] See p. 900.

[4] The cooperating laboratories for kinematic viscosity measurements were Union Oil Company of California; Standard Oil Company of New Jersey; Petroleum Refining Laboratory, Pennsylvania State College; and Tidewater Associated Oil Co. All kinematic viscosimeters were calibrated using water as the ultimate reference liquid. The viscosity of water was assumed to be 1.005 centipoises at 20 C.

[5] The cooperating laboratories for Saybolt viscosity measurements were Standard Oil Company of New Jersey, The Texas Company, Socony-Vacuum Co., and The Atlantic Refining Co.

[6] These conversions were developed by Petroleum Refining Laboratory, Pennsylvania State College; Standard Oil Company of New Jersey; Union Oil Company of California; Anglo-American Oil Co.; and A.S.T.M. (D 341 – 32 T).

Cooperative Results:

In accordance with this plan petroleum fractions with viscosities varying from 32 to 6000 Saybolt sec. (2 to 1300 centistokes) for the temperature range from 70 F. to 300 F. were submitted to the designated laboratories for viscosity determination. Both the Saybolt and kinematic viscosities of each sample were measured at the same temperatures by four laboratories, and the results obtained on each sample at each temperature were averaged for use in plotting. The average experimental kinematic value was converted to Saybolt viscosity by means of the basic averages and the deviation, in per cent, of the experimentally determined Saybolt values from these values was plotted in Fig. 1. All experimental results are given in Table I. This table also includes the data shown graphically in Figs. 1 and 2. Figure 1 illustrates

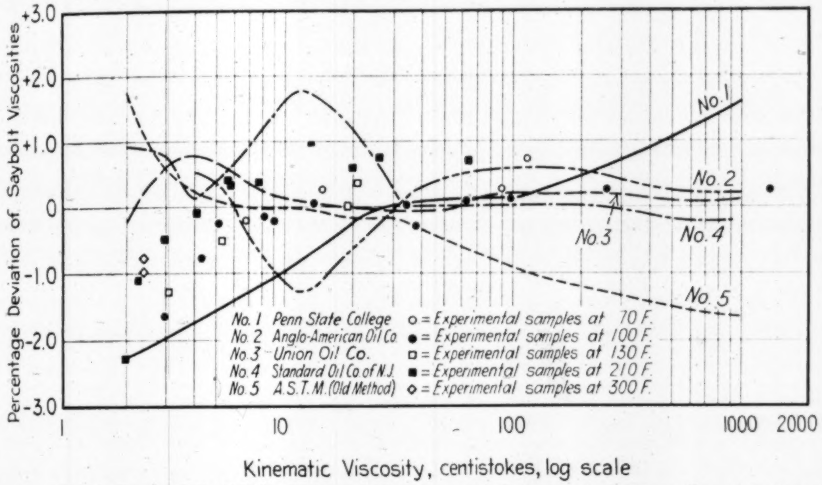

FIG. 1.—Deviation of Individual Conversions and Cooperative Experimental Results from the Average of Five Independently Derived Conversions.

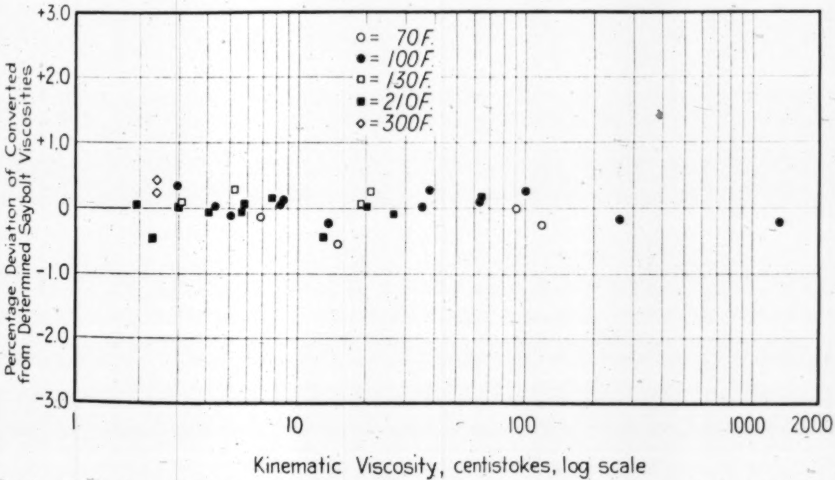

FIG. 2.—Deviation of Saybolt Viscosities Obtained by Means of New Conversion from Saybolt Viscosities Obtained Experimentally.

TABLE I.—EXPERIMENTAL DATA AND DEVIATION OF CONVERTED FROM EXPERIMENTAL SAYBOLT RESULTS.

Sample	Temperature, deg. Fahr.	Experimental Kinematic Viscosities, centistokes					Experimental Saybolt Universal Viscosities, sec.					Saybolt Viscosities By Conversion of Kinematic, sec.		Percentage Deviation of Converted from Experimental Values	
		Laboratory 1	Laboratory 2	Laboratory 3	Laboratory 4	Average	Laboratory 1	Laboratory 2	Laboratory 3	Laboratory 4	Average	By Average of 5 Conversions	By New Conversion	By Average of 5 Conversions	By New Conversion
137128-E	70	6.98	....	6.90	6.93	6.94	48.6	48.3	48.6	48.3	48.5	48.60	48.43	+0.2	−0.1
137128-C	70	14.97	....	14.86	14.88	14.90	77.4	77.0	77.2	76.8	77.1	76.92	76.68	−0.2	−0.5
137128-A	70	91.4	....	91.13	91.55	91.4	422.0	420.6	423.1	422.4	422.0	420.9	421.4	−0.3	−0.1
137128-B	70	118.6	....	118.0	118.5	118.4	547.2	545.9	551.0	545.3	547.4	545.4	545.9	−0.4	−0.3
137119	100	3.017	2.983	3.008	3.013	3.005	36.0	35.9	36.1	35.7	35.9	36.50	36.02	+1.7	+0.3
137128-E	100	4.45	....	4.438	4.426	4.440	40.5	40.6	40.5	40.3	40.5	40.85	40.50	+0.8	0.0
119000-E	100	5.22	5.16	5.21	5.20	5.20	42.9	43.0	43.2	42.7	43.0	43.12	42.94	+0.3	−0.1
137128-C	100	8.46	....	8.41	8.40	8.42	53.5	53.3	53.4	53.1	53.4	53.49	53.43	+0.2	+0.1
119000-F	100	8.88	8.80	8.85	8.85	8.85	54.85	54.95	55.0	54.5	54.8	54.91	54.89	+0.2	+0.2
137109	100	13.66	13.73	13.70	13.66	13.69	72.4	72.5	72.3	72.3	72.4	72.35	72.22	−0.1	−0.3
119000-G	100	35.25	34.86	35.20	35.18	35.12	164.0	163.7	163.8	163.2	163.7	163.71	163.74	0.0	0.0
137128-A	100	38.3	....	38.25	38.30	38.30	177.8	177.8	177.5	177.3	177.6	178.15	178.10	+0.3	+0.3
137128-B	100	45.4	....	45.28	45.21	45.30	209.5	209.1	208.8	209.6	209.3	209.95	209.7	+0.3	+0.2
A.P.I. Alpha-35	100	63.61	63.40	63.42	63.50	63.48	293.1	293.1	293.1	293.1	293.1	292.9	293.4	−0.1	+0.1
119000-H	100	100.2	99.5	100.0	99.90	99.90	459.7	459.9	461.6	460.4	460.4	459.9	461.5	−0.1	+0.2
119000-I	100	260.3	259.2	260.7	260.5	260.2	1203.0	1200.0	1205.0	1208.0	1204.0	1201.0	1202.0	−0.3	−0.2
119000-J	100	1342.0	1314.5	1340.0	1322.0	1330.0	6174.0	6084.0	6132.0	6243.0	6158.0	6142.0	6145.0	−0.3	−0.2
137128-E	130	3.105	....	3.086	3.086	3.09	36.6	36.3	36.3	36.0	36.3	36.77	36.36	+1.3	+0.2
137128-C	130	5.38	....	5.359	5.355	5.36	43.5	43.4	43.5	43.2	43.4	43.62	43.53	+0.5	+0.3
137128-A	130	19.35	....	19.32	19.35	19.34	94.9	94.9	94.5	94.4	94.7	94.7	94.74	0.0	0.0
137128-B	130	21.49	....	21.51	21.52	21.51	104.0	104.2	103.3	103.7	103.8	103.45	104.05	−0.3	+0.2
119000-A	210	1.926	1.889	1.919	1.944	1.920	32.5	32.5	32.6	32.4	32.5	33.26	32.51	+2.4	0.0
137128-C	210	2.30	....	2.291	2.293	2.29	34.0	33.9	34.1	33.8	34.0	34.37	33.84	+1.1	−0.5
137120	210	3.017	2.975	3.012	3.028	3.008	36.3	36.5	36.3	36.1	36.3	36.50	36.33	+0.6	+0.1
137103	210	4.085	4.075	....	4.090	4.083	39.6	39.7	40.0	39.6	39.7	39.75	39.70	+0.1	0.0
137128-A	210	5.66	....	5.652	5.638	5.65	44.9	44.6	45.0	44.4	44.7	44.52	44.68	−0.4	−0.1
137128-B	210	5.80	....	5.787	5.778	5.79	45.2	45.1	45.4	44.7	45.1	44.95	45.13	−0.3	+0.1
119000-B	210	7.72	7.61	7.70	7.72	7.69	51.2	51.3	51.6	51.0	51.3	51.08	51.35	−0.4	+0.1
137110	210	13.24	13.25	13.28	13.26	13.26	71.3	71.5	71.3	71.3	71.4	70.76	71.09	−0.9	−0.4
119000-C	210	20.11	19.85	20.11	20.13	20.05	98.4	98.5	98.5	98.2	98.4	97.81	98.41	−0.6	0.0
A.P.I. Beta-35	210	26.24	26.13	26.25	26.25	26.22	125.3	125.3	125.3	125.3	125.3	124.37	125.17	−0.7	−0.1
119000-D	210	63.91	64.0	64.15	64.15	64.05	299.3	295.6	296.5	299.0	297.6	295.5	298.1	−0.7	+0.2
137128-A	300	....	....	2.442	....	2.44	34.6	34.7	34.3	34.4	34.5	24.82	34.65	+0.9	+0.4
137128-B	300	....	....	2.416	....	2.42	34.5	34.8	34.3	34.3	34.5	34.76	34.58	+0.8	+0.2

the progressive deviation of the experimental results, in the low viscosity range, from the values given by the basic averages and the agreement, in the intermediate and high viscosity range, between the experimental values at 100 F. and the basic averages which were also based largely on determinations at 100 F. In order to detect any temperature effects, most of the viscosity measurements were made at 100 and 210 F. with a few at 70, 130 and 300 F.

Smooth curves were drawn through the experimental points in Fig. 1 for both the 100 and 210 F. samples. These curves then gave the percentage correction which must be applied to the basic averages at any kinematic viscosity in order to make them conform to experimental results. Using the results at 100 F. in this fashion a new table of Saybolt equivalents for 100 F. was prepared and is given in Table II. This constitutes the basis for the new conversion.

It will be observed in Fig. 1 that for a given kinematic viscosity the Saybolt equivalent varies appreciably with temperature. This variation is approximately constant at 0.7 per cent between 100 and 210 F. and on the basis of results at those two temperatures the following equation was obtained for calculating the Saybolt equivalent at any temperature from the corresponding value at 100 F. given in Table II:

TABLE II.—NEW CONVERSION OF KINEMATIC TO SAYBOLT UNIVERSAL VISCOSITY.

Kinematic Viscosity at 100 F., centistokes	Saybolt Universal Viscosity at 100 F., sec.	Kinematic Viscosity at 100 F., centistokes	Saybolt Universal Viscosity at 100 F., sec.	Kinematic Viscosity at 100 F., centistokes	Saybolt Universal Viscosity at 100 F., sec.	Kinematic Viscosity at 100 F., centistokes	Saybolt Universal Viscosity at 100 F., sec.
2	32.6	14	73.4	29	136.5	44	203.8
2.5	34.4	15	77.2	30	140.9	45	208.4
3	36.0	16	81.1	31	145.3	46	213.0
3.5	37.6	17	85.1	32	149.7	47	217.6
4	39.1	18	89.2	33	154.2	48	222.2
4.5	40.7	19	93.3	34	158.7	49	226.8
5	42.3	20	97.5	35	163.2	50	231.4
6	45.5	21	101.7	36	167.7	55	254.4
7	48.7	22	106.0	37	172.2	60	277.4
8	52.0	23	110.3	38	176.7	65	300.4
9	55.4	24	114.6	39	181.2	70	323.4
10	58.8	25	118.9	40	185.7		
11	62.3	26	123.3	41	190.2		
12	65.9	27	127.7	42	194.7		
13	69.6	28	132.1	43	199.2		

Above 70 centistokes: Saybolt seconds = centistokes × 4.620.

To obtain the Saybolt equivalent of a kinematic viscosity determined at t F. multiply the corresponding Saybolt value at 100 F. by $1 + (t - 100)\ 0.000064$; thus, 10 centistokes at 210 F. are equivalent to 58.8×1.0070 or 59.2 Saybolt sec. at 210 F.

$$\text{Saybolt seconds at } t \text{ deg. Fahr.} = 1 + (t - 100)\ 0.000064 \times (\text{Saybolt seconds at 100 F.}).$$

Thus if the Saybolt viscosity is desired for a kinematic viscosity of 10 centistokes at 210 F. the Saybolt equivalent for 10 centistokes at 100 F., given in Table II, or 58.8 sec., is multiplied by 1 + (210–100) 0.000064, or 1.0070, to give a Saybolt viscosity of 59.2 sec. at 210 F. Table II includes this equation as well as the Saybolt equivalents at 100 F., both of which comprise the new conversion method.

Accuracy:

The 32 average experimental kinematic viscosities were converted to Saybolt by means of this new conversion and the deviations of these values from the average experimental Saybolt values are shown in Fig. 2. The average deviation is 0.17 per cent while the maximum deviation is 0.5 per cent. Deviations of the individual Saybolt determinations, given in Table II, from the conversion are not shown, but in all cases they are less than 1 per cent, only a few samples showing more than 0.5 per cent deviation from the converted value.

It is quite probable that Saybolt viscosities determined on instruments having appreciable corrections will deviate more from the values obtained by conversion than is indicated above, particularly in the low viscosity range. It was for this reason that, as pointed out in the fore part of this paper, the Saybolt instruments selected for the development of this conversion were practically zero-correction instruments.

Acknowledgment:

The authors wish to thank the cooperating laboratories previously designated for their assistance in this work and to emphasize the fact that only through the excellent experimental results submitted by these laboratories has the development of this conversion been made possible.

APPENDIX IV

DIESEL-FUEL-OIL CLASSIFICATION

Manufacturers and users of Diesel engines, as well as petroleum refiners and distributors, have long felt the need for some classification differentiating types of fuel oils suitable for Diesel engines. The classification shown in Table I, covering five grades of oil, was developed by Technical Committee C on Fuel Oils (Including Diesel Fuel) of Committee D-2 on Petroleum Products and Lubricants in an attempt to supply this need. This classification was first presented in June, 1934, and has been revised as a result of suggestions since received.

TABLE I.—DIESEL-FUEL-OIL CLASSIFICATION.

Grade of Diesel Fuel	Flash Point, deg. Fahr.	Water and Sediment, per cent by Volume	Viscosity, seconds			Carbon Residue, per cent by weight	Ash, per cent by weight	Pour Point, deg. Fahr.	Sulfur, per cent by **weight**	Alkali and Mineral Acid	Ignition Quality
			Saybolt Universal (at 100 F.)		Saybolt Furol (at 122 F.)						
	Min.[a]	Max.	Min.[b]	Max.	Max.	Max.	Max.	Max.[c]			
No. 1-D.....	115 or legal	0.05	35	50	...	0.2	0.02	35	[d]	none	[e]
No. 3-D.....	150	0.1	35	70	...	0.5	0.02	35	[d]	none	[e]
No. 4-D.....	150	0.6	..	250	...	3.0	0.04	35	[d]	none	[e]
No. 5-D.....	150	1.0	..	...	100	6.0	0.08	..	[d]	none	..
No. 6-D.....	Limits for No. 6-D fuel are the same as for No. 6 oil in the Tentative Specifications for Fuel Oils (A.S.T.M. Designation: D 396 - 34 T) of the American Society for Testing Materials,[1] unless modified by special negotiations between buyer and seller.										

[a] Minimum flash point, as stated or as required by local fire regulations, Fire Underwriters or state laws.

[b] For viscosities below 35 sec. at 100 F. Saybolt Universal, other methods than that by the Saybolt Universal viscosimeter at 100 F. may be used and the results converted.

[c] Lower pour points may be specified whenever required by local temperature conditions to facilitate storage and use, although the minimum viscosity specified for Oils Nos. 1-D and 3-D must be waived whenever it is necessary to require a pour point of less than 10 F.

[d] So far as known, sulfur content need not be considered as regards combustion characteristics. However, when sulfur content is of consequence—as, for instance, in fuel for engines in intermittent service—the following limits are suggested:

Fuel No. 1-D........	1.5 per cent max.	Fuel No. 4-D........	2.0 per cent max.
Fuel No. 3-D........	1.5 per cent max.	Fuel No. 5-D........	2.0 per cent max.

[e] See Table II and discussion in Paragraph (*h*) of the text.

TABLE II.—IGNITION QUALITY.[a]

	Oil Grades		
	No. 1-D	No. 3-D	No. 4-D
Cetane number by the delay method, min.	45	35	30
Diesel index number, min................	45	30	20
Viscosity-gravity number, max...........	0.86	0.89	0.91
Boiling-point - gravity number, max......	188	195	200

[a] The values in Table II are equivalent within the limits of error of the test methods used. As the correlations between indexes and between any index and engine operation in the field are not completely established as yet, these values should be used in an approximate way only.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 882 (1934); also 1937 Book of A.S.T.M. Tentative Standards, p. 704.

It is not the intention of the committee that this classification should be used as purchase specifications; but rather that fuels purchased be correlated according to the proposed classification in order to determine its usefulness. A series of Diesel-fuel-oil specifications should evolve as a result of this program. Criticism and comment should be forwarded to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 50 West 50th Street, New York, N. Y.

METHODS OF TESTING

The properties listed in this classification are determined in accordance with the following methods of testing:

(*a*) *Flash Point.*—In absence of legal or local requirements, the minimum flash point shall be determined in accordance with the Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester (A.S.T.M. Designation: D 93) of the American Society for Testing Materials.[1]

(*b*) *Water and Sediment.*—Standard Method of Test for Water and Sediment in Petroleum Products, by Means of Centrifuge (A.S.T.M. Designation: D 96) of the American Society for Testing Materials.[2]

(*c*) *Viscosity.*—Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88) of the American Society for Testing Materials.[3]

(*d*) *Carbon Residue.*—Standard Method of Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (A.S.T.M. Designation: D 189) of the American Society for Testing Materials.[4]

(*e*) *Ash.*—Place 10 g. of the fuel oil in a weighed uncovered platinum crucible of a capacity not greater than 100 ml. and heat gently with a Bunsen burner until the oil fires and continues to burn when the flame of the burner is applied to the surface of the oil. Allow the oil to burn without further application of heat. The oil will continue to burn until practically all of it has disappeared. The time required will be about 50 min. Then place the platinum crucible in a gas or electric furnace and heat to a bright red heat until all carbon is consumed. Cool, weigh and report the residue as ash expressed as a percentage of the original 10-g. sample.

(*f*) *Pour Point.*—Standard Method of Test for Cloud and Pour Points (A.S.T.M. Designation: D 97) of the American Society for Testing Materials.[5]

(*g*) *Sulfur.*—Standard Method of Test for Sulfur in Petroleum Oils by Bomb Method (A.S.T.M. Designation: D 129) of the American Society for Testing Materials.[6]

(*h*) *Alkali and Mineral Acid.*—Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants (A.S.T.M. Designation: D 188-27 T) of the American Society for Testing Materials.[7]

(*i*) *Ignition Quality.*—According to present opinion, combustion knock in a fuel-oil-injection engine is caused by the accumulation of fuel in its cylinders prior to ignition. Even under conditions that insure ignition, the fuel may not ignite instantaneously but only after a definite delay. The longer this delay, the more fuel accumulates in the cylinders, which then burns unevenly, finally causing audible knock. If the delay is extreme, the engine will fail to operate with the fuel. It is significant that every factor which tends to aggravate knocking in a spark-ignition gasoline engine tends to suppress it in Diesel engines. Fuels of paraffin base consisting chiefly of saturated straight-chain hydrocarbons give the smoothest combustion in general, while naphthenes and aromatics burn roughly at low compression ratios. It is known that highly cracked products give trouble and also that the amount of trouble is increased as the speed of the engine is increased. Turbulence in the charge mixture reduces the delay period and turbulence is a controlling factor in engine speed. It is now generally agreed that ignition quality is one of the most important characteristics of Diesel fuels.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 897.

[2] *Ibid.*, p. 1001.

[3] *Ibid.*, p. 982.

[4] *Ibid.*, p. 849.

[5] *Ibid.*, p. 853.

[6] *Ibid.*, p. 974.

[7] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I, p. 927 (1927); also 1937 Book of A.S.T.M. Tentative Standards, p. 737.

The committee realizes the impossibility of reducing all types and designs of engines to a common rating for fuel-burning characteristics. Engine factors, such as combustion-chamber design, turbulence, spray characteristics, compression ratio, injection timing, and engine speed, vary greatly in different engine makes. Tests of ignitibility on various types of engines have no theoretical significance but have provided practical data for the types and sizes of engines on which the tests were performed. Fuel characteristics are based on a study of field-engine performance correlated with laboratory ratings of various test fuels. Ignitibility, one of the most important characteristics from the standpoint of satisfactory engine operation, has been expressed by such indices of performance as cetane number, Diesel index number, viscosity-gravity number, and boiling-point-gravity number.

CETANE NUMBER.—Cetene number was first proposed by Boerlage and Broeze of the Bataafsche Petroleum Maatschappij[1] and is the percentage of cetene in a blend of cetene and alphamethylnaphthalene which has the same ignition quality (measured in a specified engine under a definite set of engine conditions) as the fuel under test. It has been found that more reliable results may be possible when using cetane rather than cetene. For this reason, this classification substitutes the cetane number, which is determined and expressed in the same manner as the cetene number, namely, as the percentage of cetane in a blend of cetane and alphamethylnaphthalene.

DIESEL INDEX NUMBER.—Diesel Index number is calculated from aniline point and A.P.I. gravity by the following formula:

$$\text{Diesel Index Number} = \frac{(\text{A.P.I. gravity at 60 F.}) \times (\text{aniline point in deg. Fahr.})}{100}$$

Aniline Point is the lowest temperature at which equal parts by volume of freshly distilled aniline and the test sample of oil are completely miscible. It is determined by heating such a mixture in a jacketed test tube to a clear solution and noting the temperature at which turbidity appears as the mixture is cooled.

For A.P.I. Gravity see the Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (A.S.T.M. Designation: D 287) of the American Society for Testing Materials.[2]

VISCOSITY - GRAVITY NUMBER.—Viscosity - Gravity Number was first proposed by Hill and Coats in 1928 and a modification was later advanced by Moore and Kaye in 1934.[3] The number is determined by the following equation:

$$G = 1.082\,A - 0.0887 + (0.776 - 0.72A) \times \log\log(KV - 4)$$

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[1] Boerlage and Broeze, "Ignition Quality of Diesel Fuels as Expressed in Cetene Numbers," *Journal*, Soc. Automotive Engrs., July, 1932.

[2] 1937 Supplement to Book of A.S.T.M. Standards, p. 121.

[3] Moore and Kaye, "Practical Evaluation of the Ignition Characteristics of Diesel Fuel Oils," *Oil and Gas Journal*, p. 108, November 15, 1934.

where G = specific gravity at 60 F.
A = viscosity - gravity constant
KV = kinematic viscosity in millistokes at 100 F.

BOILING-POINT - GRAVITY NUMBER.—Boiling-Point - Gravity Number was first proposed by E. A. Jackson[1] in 1935. Generally speaking, the larger the number, the more naphthenic the oil, and the lower the number, the more paraffinic the oil. The number is determined by the following equation:

$$G = A + (68 - 0.703A) \log B.P.$$

where G = A.P.I. gravity in degrees
A = Boiling-point - gravity constant
$B.P.$ = 50 per cent boiling point on A.S.T.M. distillation curve in degrees Cent.

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[1] E. A. Jackson, "Boiling-Point - Gravity Constant is Index of Lubricating Oil Characteristics," *Oil and Gas Journal*, March 21, 1935.

REPORT OF SECTIONAL COMMITTEE
ON
PETROLEUM PRODUCTS AND LUBRICANTS
A.S.A. PROJECT: Z11

The organization of this new Sectional Committee on Petroleum Products and Lubricants was effected at a meeting held on January 24, 1936, in New York City. A further meeting was held on July 1, 1936, during the annual meeting of the A.S.T.M., at which the permanent officers were elected and other details of organization completed. Two meetings have been held subsequently. The sectional committee functions under the procedure of the American Standards Association with the A.S.T.M. as sponsor, the approved scope of the sectional committee being as follows:

Scope.—Specifications, methods of test and nomenclature relating to crude petroleum and petroleum products (products derived in whole or in large part from petroleum) other than organic chemicals, products used medicinally, and road, paving, waterproofing, and electrical insulating materials.

For a number of years A.S.T.M. Committee D-2 on Petroleum Products and Lubricants has been developing standard methods of test for petroleum products and lubricants and has published critical studies of the significance of these tests in relation to service performance of the products. All of this work is fundamental and essential in paving the way for the development of specification requirements for petroleum products. Many agencies, trade associations and industrial companies have indicated an increasing interest in specification work and in order to plan and coordinate standardization work in this field, steps were taken to organize the sectional committee. Under A.S.A. procedure, two sectional committees were functioning in this field, dealing respectively, with nomenclature and methods of testing, and with specifications for fuel oils. The work of these two former committees is now covered by the new sectional committee which is known as A.S.A. Project Z11. This is the same designation applying to the former sectional committee dealing with test methods.

The personnel of the committee is made up of representatives of 32 national technical organizations interested in petroleum products and lubrication, the classification of the personnel being as follows: producers 12, consumers 22, general interest members 7, making a total of 41 members.

In order that complete information might be available to members of the sectional committee with respect to work in progress, both of a standardization and research nature in the field of the sectional committee, summaries of work under way in the field of petroleum products are in course of preparation. These relate to lubricating oils, to greases, to gasoline, both motor and aviation, and to fuel oils for furnaces and for Diesel engines. It is hoped later to make these compilations available through publication.

A Special Committee was appointed to prepare a general discussion with respect to the physical properties of lubricants under conditions of boundary lubrication, such as, oiliness, film strength, etc. The chairman of the Special Committee is J. C. Geniesse.

Another Special Committee under the chairmanship of L. C. Beard, Jr., has investigated the possibility of setting up a general viscosity classification of lubricating oils. After much study, this group has just rendered the following report to the sectional committee, which it has approved:

> "It is the opinion of the committee that at present there appears to be no marked need for a viscosity classification of industrial lubricants and, under the circumstances, it appears reasonable to postpone any action on such a classification until such time as requests from the users of oil are more emphatic. It would be extremely difficult to make an industrial lubricating oil classification which would be satisfactory to both consumers and producers. Such a classification, if written so as to reduce the number of grades required and to simplify specification writing, would undoubtedly in most instances be too broad to serve any useful purpose."

The matter of specifications for motor gasolines was delegated to A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Through its Technical Committee A on Gasoline, this committee has given considerable study to the formulation of such specifications and has submitted to the A.S.T.M. during the year Tentative Specifications for Gasoline (D 439 – 37 T) which were subsequently approved and published by the Society.[1] These specifications are now before the sectional committee.

In line with its function as a reviewing body, the sectional committee has taken action on 19 A.S.T.M. methods for reference to the American Standards Association for approval either as American standard or American tentative standard.

Respectfully submitted on behalf of the sectional committee,

T. A. BOYD,
Chairman.

R. E. HESS,
Secretary.

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[1] See p. 872.

REPORT OF COMMITTEE D-3
ON
GASEOUS FUELS

In the first annual report[1] of this committee presented last year a history of the formation of the committee, its objectives and scope were given. Formation of subcommittees was completed last year and their work inaugurated. This second annual report is confined, therefore, to a record of activities during the past year. Committee work has been aggressively pushed and many preliminary considerations disposed of. The membership of the committee now comprises a total personnel of 30 members, consisting of 11 producers, 10 consumers, and 9 representing general and scientific interests.

Two meetings of Committee D-3 and its seven subcommittees have been held since June of last year. At the first meeting in Atlantic City, N. J., on October 27, 1936, the former officers of the main committee were unanimously re-elected.

A second meeting of Committee D-3 was held in Chicago, Ill., on March 2, 1937. Each subcommittee chairman presented a brief review of the progress of his group on the subjects assigned. Consideration was given to the need of conducting research work to standardize various test methods and ways and means of securing cooperation from interested organizations. Studies and discussions have further emphasized the need for and value of standardized procedures and test methods for gaseous fuels. When carried to completion along the lines now planned, the results should be of lasting benefit to the gas industry, to scientific institutions and to the users of gaseous fuels in general.

All detailed work has been assigned to the seven interested subcommittees and brief reports on their activities follow.

Subcommittee I on Collection of Gaseous Samples (S. S. Tomkins, chairman; F. E. Vandaveer, secretary).—The plan of procedure decided upon for the work of this subcommittee consists of a study of three separate items: (1) definitions, (2) fundamental details, and (3) existing methods in practice. A section has been formed to prepare standard definitions relating to sampling. A questionnaire on this subject is now being answered by the section members. On the subject of fundamental details, the chairman is preparing material for committee consideration covering the following items:

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 422 (1936).

1. Importance of sampling procedure,
2. Classification of samples,
3. Sampling apparatus,
4. Locations for securing samples,
5. Physical arrangement of sampling apparatus, and
6. Containers for sample storage.

A questionnaire on methods now in common use in industry has been submitted to all members of the subcommittee. Replies to this questionnaire will be studied and reviewed at the next meeting of the subcommittee.

Subcommittee II on Measurement of Gaseous Samples (H. S. Bean, chairman).—This subcommittee has approved by letter ballot and referred to the main committee for consideration as tentative standards, the following:

1. A statement of the units in which gaseous samples are to be measured,
2. A statement of standard conditions for the measurement of gaseous samples, and
3. A definition of a unit of volume.

Tests that have been made in connection with the work of this subcommittee have shown that the use of a fractional revolution of a laboratory wet-gas meter may introduce errors as high as 10 to 12 per cent.

This group is sponsoring tests to determine the effects upon the accuracy of gas measurements brought about by changes in its relative humidity.

Subcommittee III on Determination of Calorific Value of Gaseous Fuels (R. B. Harper, chairman; K. R. Knapp, secretary).—Work of this subcommittee has been sectionalized under three leaders, to cover (1) preparation of standard definitions, (2) study of fundamentals covering standard equipment, with the thought of establishing both referee and routine methods of heating value determinations, and (3) investigation of existing practices now followed throughout the industry for the determination of calorific values.

Progress is reported in all of these sections. Authorities and references have been consulted and proposed definitions have been prepared, discussed, and criticisms thereon referred to the appropriate section for review. Studies are being made of fundamentals concerning referee and routine methods of calorific value determinations. Many industrial, Governmental, and educational organizations have been and are being requested by letter and questionnaire to furnish detailed information on existing practices.

Consideration is also being given to the design and construction of a special calorimeter capable of giving highly accurate heating value data under controlled conditions, and to research work on existing equipment with various gases in comparison with the special calorimeter.

Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels (C. D. Ulmer, chairman; F. O. Suffron, secretary).—Division of work of this subcommittee has been assigned to four sections: Section A on Survey of Methods and Bibliography; Section B on Definitions; Section C on Fundamental Ideas Relating to the Subject; and Section D on Precise Laboratory Methods. Proposed definitions have been prepared and are to be given further study before being submitted to letter ballot of the committee. Many factors affecting precise laboratory methods have been discussed in the two meetings held this year and various factors clarified.

Subcommittee V on Determination of Impurities of Gaseous Fuels (E. F. Pohlmann, chairman; S. J. Modzikowski, secretary).—Impurities have been tentatively defined by this subcommittee, as follows:

> Impurities in gaseous fuels are substances which under certain specific conditions may be deemed undesirable from the standpoint of transmission, distribution, and utilization of the gas in question. Substances often treated as normal constituents of gaseous fuels may under specific conditions and at specific times be considered as impurities. In general, there are two classes of constituents in fuel gases, namely, (1) those about which there is no question as to their desirability, and (2) those which under specific conditions are not desirable. Specific conditions are functions of specific cases and therefore cannot be defined. Consequently, it is possible for a constituent of a fuel gas to be desirable at one time and undesirable at another time.

It was decided to formulate methods for the determination of the following impurities in the order named: (1) hydrogen sulfide and other sulfur compounds, (2) tar fog and condensible oils and vapors, (3) naphthalene, (4) gums, (5) ammonia, (6) oxides of nitrogen, (7) dust, (8) cyanogen, (9) oxygen, and (10) iron carbonyl. Other constituents may be considered later.

A canvass of the committee membership and others interested in this work as to the methods in common use for the determination of the above named impurities is now being conducted.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels (A. W. Gauger, chairman; F. O. Suffron, secretary).—This subcommittee's section on definitions has completed a tentative draft which is ready for final action by the entire subcommittee. A complete résumé of methods for determining water-vapor content of gases was presented to the subcommittee by the chairman at the last meeting. Full discussion of the various items relating to this subject has taken place at the two meetings held this year. Preliminary work has therefore practically been completed and future activity will be directed toward a research study on methods and apparatus.

This subcommittee is also sponsoring some experimental work on one of its problems which is being carried out under the chairman's direction at Pennsylvania State College.

Subcommittee VII on Complete Analysis or Chemical Composition of Gaseous Fuels (Martin Shepherd, chairman; F. E. Vandaveer, secretary).—The program of work adopted by this subcommittee is as follows:

1. Preparation of a list of gaseous fuels for which standard apparatus and methods are to be prescribed,
2. Selection of analytical tolerances (reproducibility, and accuracy) for the determination of the constituents of each type of gaseous fuel, which will satisfactorily meet the requirements prescribed by the uses of the analytical data,
3. A study of the suitability of apparatus and methods by means of analyses of samples of known composition, and
4. Approval of all reasonable apparatus and methods which can be shown according to item 3 to meet the requirements prescribed in item 2.

The subcommittee has completed items 1 and 2 as noted above. These data are considered tentative at present and may be altered if further need develops.

A complete list of apparatus and methods in present use is now being prepared by Subcommittee VII, with the object of selecting the more suitable to be employed in experimental studies.

This report has been submitted to letter ballot of the committee, which consists of 30 members; 30 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. FIELDNER,
Chairman.

R. M. CONNER,
Secretary.

R. B. HARPER,
Vice-Chairman.

REPORT OF COMMITTEE D-4
ON
ROAD AND PAVING MATERIALS

Committee D-4 on Road and Paving Materials held two meetings during the past year, in Atlantic City, N. J., on July 2, 1936, and in Chicago, Ill., on March 2, 1937.

Subsequent to the 1936 annual meeting, Committee D-4, in cooperation with Committee C-9 on Concrete and Concrete Aggregates, presented to the Society through Committee E-10 on Standards the following two proposed tentative methods of test:

Tentative Methods of:

Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 36 T), to replace the Standard Method of Test for Apparent Specific Gravity of Coarse Aggregates (D 30 – 18) and the Tentative Method of Test for Apparent Specific Gravity of Coarse Aggregates in a Saturated Condition (C 86 – 31 T).

Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 36 T), to replace the Standard Methods of Test for Apparent Specific Gravity of Sand, Stone and Slag Screenings and Other Fine Non-Bituminous Highway Materials (D 55 – 25) and the Standard Method of Test for Approximate Apparent Specific Gravity of Fine Aggregate (C 68 – 30).

These proposed methods were accepted[1] for publication as tentative on August 26, 1936, and appear in the 1936 *Proceedings*.[2] The four methods which were superseded by these two new tentative standards were accordingly discontinued.

During the past year Subcommittee B-21 on Solubility Tests (H. M. Milburn, chairman) has conducted a series of cooperative tests on salt-asphalt mixtures. This work was undertaken because some asphaltic products contain a small amount of salts which it is desirable to determine so that no portion of them will be classified as insoluble organic material, and the Standard Method of Test for Determination of Bitumen (D 4 – 27) does not provide for accurate identification of material insoluble in carbon disulfide.

The procedure used in conducting these cooperative tests was as follows:

Procedure.—For the analysis, use method No. 1 of the Standard Method of Test for the Determination of Bitumen (A.S.T.M. Designation: D 4) of the American Society for Testing Materials[3] with the following changes and additions:

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[1] In submitting these tentative methods to Committee E-10 on Standards, Committee D-4 reported results of the letter ballot vote as follows: Of a total membership of 102, 67 members returned their ballots, of whom 61 voted affirmatively, 0 negatively, and 6 members marked their ballots "not voting."

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 805 and 808 (1936); also 1936 Book of A.S.T.M. Tentative Standards, pp. 511 and 514.

[3] 1936 Book of A.S.T.M. Standards, Part II, p. 1041.

(*a*) Weigh from 4 to 5 g. of the asphalt-salt mixture into a 125-ml. Erlenmeyer flask and dissolve and filter according to Method D 4 – 27, but dry the crucible and content overnight in a desiccator instead of in an oven at 100 to 125 C.

NOTE.—The samples were thoroughly stirred in their preparation but as there may be some settlement of the added salt the samples should be carefully heated and thoroughly stirred just prior to sampling for analysis.

(*b*) After the Gooch crucible has been weighed to determine the weight of the total carbon disulfide insoluble material on the felt, wash the contents of the crucible with hot distilled water, using suction to facilitate filtration, until the filtrate gives no test for chlorides with 0.1 *N* $AgNO_3$ solution. Dry the crucible in an oven at 110 C. to constant weight. Cool in desiccator, and weigh. Then ignite crucible, cool in a desiccator, and weigh.

TABLE I.—TABULATION OF RESULTS OF PERCENTAGES OF WATER-SOLUBLE SALTS OBTAINED ON ASPHALT-SALT MIXTURES.

Sample Designations	Percentage of Salt Present as Reported by Cooperators					Calculated Salt Content, per cent
	C. N. Forrest	D. H. Jenks, Jr.	K. G. Mackenzie	H. W. Skidmore	H. M. Milburn[a]	
A	.....	.....	....	.....	0.018	.....
N.5A	.....	.....	....	.....	0.497	0.515
N1A	.....	.....	....	.....	1.009	1.024
M.5A	.....	.....	....	.....	0.476	0.520
M1.5A	.....	.....	....	.....	1.460	1.618
B	0.013	.....	....	.....	0.018	.....
N.5B	0.475	.....	....	.....	0.484	0.515
N1B	0.980	.....	....	.....	1.031	1.024
M.5B	0.465	.....	....	.....	0.511	0.520
M1.5B	1.373	.....	....	.....	1.514	1.618
	1.402					
C	.....	0.047	....	.....	0.018	.....
N.5C	.....	0.498	....	.....	0.481	0.515
N1C	.....	1.01	....	.....	1.011	1.024
M.5C	.....	0.450	....	.....	0.494	0.520
M1.5C	.....	1.410	....	.....	1.560	1.618
D	.....	.....	0.04	.....	0.018	.....
N.5D	.....	.....	0.44	.....	0.486	0.515
N1D	.....	.....	0.92	.....	1.028	1.028
M.5D	.....	.....	0.45	.....	0.489	0.520
M1.5D	.....	.....	1.31	.....	1.525	1.618
E	.....	.....	....	0.032	0.018	.....
N.5E	.....	.....	....	0.342	0.513	0.515
N1E	.....	.....	....	0.861	1.044	1.028
M.5E	.....	.....	....	0.495	0.520	0.520
M1.5E	.....	.....	....	1.294	1.474	1.618

[a] Samples designated as A, B, C, D, and E represent samples of asphalt cement used in the preparation of the asphalt-salt mixtures and the reported results for water-soluble salt for samples A, B, C, D, and E were made on this asphalt cement and not on individual samples.

The prefixes N.5, N1, M.5, and M1.5 represent respectively samples prepared individually to contain 0.5 per cent and 1.0 per cent of sodium chloride, and 0.5 per cent and 1.5 per cent of magnesium chloride ($MgCl_2 \cdot 6H_2O$).

(*c*) Evaporate carbon disulfide filtrate in a silica dish and ignite to bright red heat, cool in a desiccator, and weigh to determine inorganic material that may have passed through the felt. If such material is present test it for the presence of chlorides.

Report.—The reported results should include the following:

(*a*) Percentage of organic and inorganic carbon disulfide insoluble material retained on the felt. (Report the percentage of each).

(*b*) Percentage of water soluble material.

(*c*) Percentage of organic and inorganic water insoluble material retained on the felt. (Report the percentage of each.)

(*d*) Percentage of inorganic material in the carbon disulfide filtrate; also report if chlorides are present in carbon disulfide filtrate.

This may serve as a basis for recommending to the Society, some time in the future, a new method of test on this subject.

The results obtained in the cooperative tests are given in Table I. The samples which were sent to the cooperating members were prepared in the laboratory of the U. S. Bureau of Public Roads by adding 0.5 per cent and 1 per cent by weight of sodium chloride; 0.5 per cent and 1.5 per cent of magnesium chloride ($MgCl_2 \cdot 6H_2O$) to an asphalt. Each sample was prepared individually. In the tabulation, the letters A, B, C, D, and E are the set identification designations. The prefixes N.5, N1, M.5, and M1.5 represent samples prepared to contain 0.5 per cent and 1.0 per cent of sodium chloride, and 0.5 per cent and 1.5 per cent of magnesium chloride ($MgCl_2 \cdot 6H_2O$), respectively.

In the cooperative tests. other results than the percentages of water-soluble salts were reported. However, the only results that the subcommittee has considered and approved for publication as informational data are the determinations of the percentages of water-soluble salts.

RECOMMENDATIONS AFFECTING STANDARDS

I. Proposed Tentative Standards:

Proposed Tentative Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine.—The committee recommends that this method, as appended hereto,[1] be accepted for publication as tentative. The method is substantially the same as that published for information in the 1935 annual report[2] of the committee. The original description has, however, been slightly revised as the result of further study.

Proposed Tentative Specifications for Standard Sizes of Coarse Aggregates for Highway Construction.—It is recommended that these specifications, as appended hereto,[3] be accepted for publication as tentative to replace the following tentative specifications which are accordingly recommended to be withdrawn:

Tentative Specifications for Commercial Sizes of Broken Stone and Broken Slag for Highway Construction (D 63 – 23 T),[4] and

Tentative Specifications for Commercial Sizes of Sand and Gravel for Highway Construction (D 64 – 20 T).[5]

II. Proposed Revision of Standard:

Standard Specifications for Materials for Cement Mortar Bed for Brick, Stone Block, Wood Block, Asphalt Block and Other Block Pavements (D 58 – 24).[6]—In order to bring these specifications more closely in line with present practice, the committee recommends that the following minor revision be adopted immediately. Accordingly, the necessary nine-tenths vote at the annual meeting is requested in order that this revision may be referred to letter ballot of the Society:

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[1] See p. 801. An additional revision presented at the annual meeting, see Summary of Proceedings, p. 9, has been incorporated in the method.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 350 (1935).

[3] See p. 918.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 709 (1923); also 1936 Book of A.S.T.M. Tentative Standards, p. 719.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. XX, Part I, p. 696 (1920); also 1936 Book of A.S.T.M. Tentative Standards, p. 713.

[6] 1936 Book of A.S.T.M. Standards ,Part II, p. 1015.

Section 2.—In Paragraph (*a*) change the table of grading requirements for the sand to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

Total passing [¼-in. screen] *a No. 4 sieve*........................100 per cent
Total passing a No. [10] *8* sieve......................not less than 90 per cent

In Paragraph (*b*) substitute for the method of mechanical analysis now specified a reference to the new method of mechanical analysis of aggregates when the latter method, which is now being prepared under the joint jurisdiction of Committees C-9 and D-4, is issued as tentative by the Society.

Section 3.—In Paragraph (*a*) change the first two sentences to read as follows by the addition of the italicized figures and the omission of those in brackets:

> The sand shall be passed through a No. [10] *8* sieve and that portion retained by the No. [10] *8* sieve shall be discarded. The portion passing a No. [10] *8* sieve shall be mixed with portland cement conforming to the requirements of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 9) of the American Society for Testing Materials, in the proportion of 1 part cement to 3 parts of sand, by weight, according to the standard method of making 1:3 mortar briquets.

III. Proposed Revision of Tentative Standard:

Tentative Method of Test for Abrasion of Gravel (*D 289 – 28 T*).[1]—To meet certain criticisms and to bring this method in line with present practice, Committee D-4 recommends that it be revised as appended hereto[2] and continued as tentative.

IV. Proposed Adoption of Tentative Standard as Standard:

Tentative Method of Test for Determination of Amount of Material Finer than No. 200 Sieve in Aggregates (*C 117 – 35 T*).[3]—This method is under the joint jurisdiction of Committee D-4 and Committee C-9 on Concrete and Concrete Aggregates. In practice, it has largely replaced the Standard Method of Test for Quantity of Clay and Silt in Gravel for Highway Construction (D 72 – 21)[4] for which Committee D-4 is responsible, and the Standard Method of Decantation Test for Sand and Other Fine Aggregates (D 136 – 28),[4] which is under the joint jurisdiction of Committees C-9 and D-4. Therefore, Committee D-4 joins with Committee C-9 in recommending that Tentative Method C 117 – 35 T be approved for reference to letter ballot of the Society for adoption as standard to replace the Standard Methods D 72 – 21 and D 136 – 28, which are recommended to be withdrawn.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part I, p. 940 (1928); also 1936 Book of A.S.T.M. Tentative Standards, p. 738.

[2] See p. 921. An additional revision presented at the annual meeting, see Summary of Proceedings, p. 10, has been incorporated in the method.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 832 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 488.

[4] 1936 Book of A.S.T.M. Standards, Part II, pp. 1050 and 1051, respectively.

V. Withdrawal of Standard:

Standard Form of Specifications for Certain Commercial Grades of Broken Stone (D 35 – 18).[1]—The committee recommends the withdrawal of this standard. This will conform with the recommendation presented earlier in this report, proposing that the related Tentative Specifications for Commercial Sizes of Broken Stone and Broken Slag for Highway Construction (D 63 – 23 T) and for Sand and Gravel for Highway Construction (D 64 – 20 T) be discontinued.

Tentative Specifications for Concrete for Pavements (D 366 – 33 T).[2]—The committee also recommends the withdrawal of these tentative specifications as being essentially construction specifications and therefore somewhat outside the province of the Society.[3] This recommendation for discontinuing these specifications is presented with the understanding that this subject will be carried on by the American Concrete Institute.

VI. Tentative Standards Continued as Tentative:

In connection with the tentative standards for which it is responsible and which have been published for three or more years without revision, Committee D-4 regrets that it is unable at this time to recommend their adoption as standard. It hopes, however, to include definite recommendations regarding them in its next annual report.

At the request of Committee D-15 on Thermometers and Laboratory Glassware, Committee D-4 has approved the inclusion of the following clause as an editorial change in the requirements for high- and low-distillation thermometers:

> For purposes of calibration and certification one or two division markings should be placed below the zero on the Centigrade scale thermometer.

The committee has also approved the following editorial revisions in the Standard Methods of Chemical Analysis of Calcium Chloride (D 345 – 34):[4]

Section 1.—Change the tenth sentence from its present form: namely,

> Dissolve the precipitate with 10 to 15 ml. of 0.25 N H_2SO_4, heat to about 80 C. and titrate to a pink color with 0.1 N $KMnO_4$.

to read as follows:

> Dissolve the precipitate with 10 to 15 ml. of a solution of 1 part H_2SO_4 and 4 parts water, heat to about 80 C. and titrate to a pink color with 0.1 N $KMnO_4$.

In the formula following this section change the denominator of the fraction to read as follows by the addition of the italicized word: "Weight of *original* sample in grams."

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1113.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 764 (1933); also 1936 Book of A.S.T.M. Tentative Standards, p. 694.

[3] This recommendation was approved by the Society at the annual meeting, see Summary of Proceedings, p. 11.

[4] 1936 Book of A.S.T.M. Standards, Part II, p. 1046.

Section 3.—Divide the second paragraph of this section into two paragraphs beginning the new third paragraph with the present sixth sentence which reads: "Finally, filter through a dry retentive filter paper, discarding the first 10 ml. and refiltering if cloudy."

Omit from the new third paragraph the second sentence, which reads as follows:

> Add a few milliliters of $(NH_4)_2C_2O_4$, heat to boiling, and filter.

Committee D-4 joins with Committee D-8 on Bituminous Waterproofing and Roofing Materials in recommending that Committee E-1 on Methods of Testing give consideration to the following changes in the Tentative Method of Test for Softening Point by Ring-and-Ball Apparatus (E 28 – 36 T):

1. To avoid confusion with present Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (D 36 – 26), change the name of of the test to read: "Method of Test for Softening Point by Tapered Ring Apparatus."
2. Remove or change the requirement in Section 3 that a sample shall "not be heated more than 30 C. (54 F.) above its softening point, in the case of materials having a softening point below 121 C. (250 F.) . . . or more than 50 C. (90 F.) . . . materials with softening points above 121 C. (250 F.)."

The change in Section 3 is suggested since certain asphaltic materials require greater differences than these between the softening point and pouring temperature.

The committee further recommends that wide publicity be given to the fact that Method E 28 – 36 T is essentially a new method which gives somewhat different results from those obtained by the Standard Method D 36 – 26. Committee D-4 does not favor the withdrawal of Standard Method D 36 – 26 nor the adoption as standard of Method E 28 – 36 T until these recommendations are adopted and Method E 28 – 36 T is acceptable to the members of the committee.

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 100 members; 70 members returned their ballots, the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. PROPOSED TENTATIVE STANDARDS			
Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine	57	1	12
Specifications for Standard Sizes of Coarse Aggregate for Highway Construction to replace Specifications D 63 – 23 T and D 64 – 20 T	56	1	12
II. REVISION OF STANDARD			
Specifications for Materials for Cement Mortar Bed for Brick, Stone Block, Wood Block, Asphalt Block and Other Block Pavements (D 58 – 24), immediate adoption	56	2	12
III. REVISION OF TENTATIVE STANDARD			
Method of Test for Abrasion of Gravel (D 289 – 28 T)	55	2	13
IV. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Method of Test for Determination of Amount of Material Finer than No. 200 Sieve in Aggregates (C 117 – 35 T), to replace Standard Methods D 72 and D 136	62	0	7
V. WITHDRAWAL OF STANDARD			
From of Specifications for Certain Commercial Grades of Broken Stone (D 35 – 18)	58	0	11

This report has been submitted to letter ballot of the committee which consists of 100 members; 70 members returned their ballots, of whom 67 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

F. C. LANG,
Chairman.

PRÉVOST HUBBARD,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting Committee D-4 on Road and Paving Materials presented to the Society through Committee E-10 on Standards a proposed tentative revision of the Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (D 61 – 24). This revision was accepted by Committee E-10 for publication as tentative on October 11, 1937, and appears on p. 1321.

REPORT OF COMMITTEE D-5

ON

COAL AND COKE

Committee D-5 on Coal and Coke held two meetings during the past year; one on July 1, 1936, at the annual meeting of the Society in Atlantic City, N. J., and the other on March 1, 1937, at the spring group meetings of A.S.T.M. committees in Chicago, Ill.

A new subcommittee, under the chairmanship of Mr. C. C. Russell and designated as Subcommittee XV on Plasticity and Swelling of Coal, has been formed to investigate methods for testing plasticity and swelling of coal when heated, in connection with the use of coal for coke-making and as affecting its burning characteristics under fuel bed conditions. Swelling or expansion of coal is important in the selection of coals for coke-making as strongly swelling coals cause injury to the walls of the ovens.

During the year, the second edition of the compilation of methods of test, specifications, and definitions sponsored by Committee D-5 was issued by the Society as a special publication entitled A.S.T.M. Standards on Coal and Coke.

It is with regret that the committee records the death of Mr. F. R. Wadleigh who was active in the work of the committee. The membership of the committee now totals 43, of whom 16 are classified as consumer, 8 as producer, and 19 as general interest members.

RECOMMENDATIONS AFFECTING STANDARDS

I. Proposed Tentative Standards.—The committee recommends that the proposed Tentative Method of Drop Shatter Test for Coal and Method of Tumbler Test for Coal, as appended hereto,[1] be accepted for publication as tentative. The shatter test is suitable for testing both a standard size of different coals and for testing different sizes of the same coal, and the tumbler test method is intended for determining the relative friability of a particular size of lump coal.

II. Proposed Revisions of Tentative Standards.—The committee recommends that the Tentative Methods of Test for Grindability of Coal by the Ball-Mill Method (D 408 – 35 T)[2] and for Grindability of Coal by the Hardgrove-Machine Method (D 409 – 35 T)[2] be revised as appended hereto[3] and continued as tentative. The revisions are based on further experimental work and criticisms received since the two methods were first published as tentative in 1935.

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[1] See pp. 820 and 827.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, pp. 854, 857 (1935); also 1936 Book of A.S.T.M. Tentative Standards, pp. 532, 535.

[3] See pp. 810 and 815.

III. Proposed Tentative Revisions of Standards.—The committee recommends the revisions as appended hereto[1] in the Standard Method of Sampling Coal (D 21 – 16) and the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 33), for publication as tentative. The proposed modifications are intended to provide a means for mechanical crushing and reduction of gross samples of coal and to specify the minimum amount of minus 4 mesh coal to constitute a laboratory sample. The proposed revisions of Methods D 271 change the quantity of the laboratory sample of minus 4 mesh coal from 10 lb. to 15 lb. to conform with the revision of Method D 21. The tolerances for check determinations of volatile matter are revised to include tolerances for anthracite, semi-anthracite, subbituminous coal, peat, and chars.

IV. Adoption as Standard of Tentative Revision of Standard.—As a tentative revision of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 33), the committee presented last year[2] a revision of the modified procedure for the determination of volatile matter in fuels which require a preliminary heat treatment to prevent the too rapid evolution of moisture and gases. The committee now recommends that this tentative revision be approved for reference to letter ballot of the Society for adoption as standard.

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 43 members, the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. PROPOSED TENTATIVE STANDARDS			
Method of Drop Shatter Test for Coal	38	0	5
Method of Tumbler Test for Coal	38	0	5
II. PROPOSED REVISION OF TENTATIVE STANDARDS			
Method of Test for Grindability of Coal by the Ball-Mill Method (D 408 – 35 T)	25	0	8
Method of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 – 35 T)	23	0	10
III. PROPOSED TENTATIVE REVISION OF STANDARDS			
Method of Sampling Coal (D 21 – 16)	32	0	1
Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 33)	32	0	1
IV. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 33)	33	0	0

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[1] See pp. 1325 and 1326.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1206 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 1344.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Methods of Testing (W. A. Selvig, chairman).—In the report of Committee D-5 as presented last year, reference was made to experiments to find a satisfactory inert material for the agglutinating value test for coal. Tests made on different lots of silicon carbide (carborundum) show that this material is sufficiently uniform for use as the inert material. The method is not yet ready for recommendation as a proposed tentative standard because of difficulty in obtaining check results among different laboratories showing that the technique of the test procedure requires further investigation.

Continued check determinations by different laboratories of coal-ash fusibility by the Barrett coal-ash fusion furnace and the gas furnace specified in the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 33) have shown that these results check well within the tolerances as given in the standard methods. Committee D-5 has instructed Subcommittee I to prepare a revision of the standard method to include the Barrett coal-ash fusion furnace. In this revision, it is proposed to give specific furnace requirements and to include names of commercial furnaces that on trial have been found to give satisfactory results.

Subcommittee II on Nomenclature and Definitions (H. C. Porter, chairman).—Upon recommendation of Subcommittee II, the Tentative Definition of the Term Coke (D 121 – 30 T) is being continued as tentative, pending further consideration by Committee E-8 on Nomenclature and Definitions, to which the matter of a revised definition for this term has been referred for reconcilement of the differences in opinion thereon.

The Tentative Definitions of the Terms Gross Calorific Value and Net Calorific Value of Fuels (D 407 – 35 T) are being continued as tentative to afford further opportunity for criticism.

Subcommittee VII on Pulverizing Characteristics of Coal (John Van Brunt, chairman).—A meeting of the subcommittee was held in New York City, on October 14, 1936, at which time extensive revisions were made in the Tentative Methods of Test for Grindability of Coal by the Ball-Mill Method (D 408 – 35 T) and for Grindability of Coal by the Hardgrove-Machine Method (D 409 – 35 T). These modifications, which have been incorporated in the revised methods appended hereto,[1] are the result of further investigative work and criticisms received since publication of the tentative methods. In the case of the Hardgrove-machine method, sieving at the end of the test is greatly shortened by the elimination of all but one sieve in size testing the ground coal. The formula given in the revisions of this method for calculating grindability from the weight of sample ground to pass a 74-micron (No. 200) sieve was derived from a large number of coals by plotting grindability indexes determined by the procedure as published against grams of coal ground to pass a 74-micron (No. 200) sieve.

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[1] See pp. 810 and 815.

Subcommittee XI on Coal Friability (R. E. Gilmore, chairman).—This subcommittee completed the task of formulating methods of test for friability of coal or its resistance to breakage on handling. These two methods entitled proposed Tentative Method of Drop Shatter Test for Coal and Method of Tumbler Test for Coal, appended hereto,[1] are being recommended for publication as tentative as mentioned earlier in this report. The experimental work upon which the methods are based is given in footnote references included in the methods.

Subcommittee XIII on Mechanical Sampling and Reduction of Samples (J. B. Morrow, chairman).—The proposed tentative revisions in the Standard Method of Sampling Coal (D 21 – 16) and the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 33), as appended hereto[2], were prepared by this subcommittee. The revision of Method D 21 for the sampling of coal provides a procedure for the reduction of gross samples of coal by means of mechanical crushers and riffle samplers. This provides a method much less laborious than the standard hand method and is probably of greater accuracy because of the elimination of personal errors inherent in hand methods of reducing coal samples. Mechanical methods of reducing coal samples are now used extensively by those having much coal sampling to perform, so there is need for a standardized procedure. The recommendation that minus 4 mesh coal be not divided further than 15 lb. is based on experimental work[3] showing an increase of sampling errors with a decrease in the size of the sample.

This report has been submitted to letter ballot of the committee, which consists of 43 members; 33 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. FIELDNER,
Chairman.

W. A. SELVIG,
Secretary.

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[1] See pp. 820 and 827.

[2] See pp. 1325 and 1326.

[3] J. A. Younkins, "Notes on the Collection and Reduction of Coal Samples," *Mineral Industries*, The Division of Mineral Industries Extension, Pennsylvania State College, Vol. 6, No. 1, pp. 3–4; No. 2, p. 2 (1936).

REPORT OF SECTIONAL COMMITTEE
ON
CLASSIFICATION OF COALS

A.S.A. PROJECT: M 20

The Sectional Committee on Classification of Coals, functioning under the procedure of the American Standards Association, held one meeting during the past year, in New York City, on February 17, 1937.

The committee records with regret the death of Mr. F. R. Wadleigh, who took an active part in the work of the committee and served as chairman of the Technical Committee on Marketing Practice. W. R. Chedsey has been appointed the representative of the Coal Mining Institute of America, *vice* W. E. Fohl, resigned. D. A. Newhall was appointed as a representative of the National Coal Association, *vice* W. H. Cunningham, resigned.

RECOMMENDATIONS AFFECTING STANDARDS

Adoption of Tentative Standards as Standard.—The committee recommends that the Tentative Specifications for Classification of Coals by Grade (D 389 – 34 T)[1] and Specifications for Classification of Coals by Rank (D 388 – 36 T)[2] with revisions as given below be approved for reference to letter ballot of the Society for adoption as standard.

In the case of Specifications D 389, the changes have been incorporated in the completely revised specifications which are appended hereto.[3] The modifications in these specifications cover the inclusion of size designations and the addition of a footnote to Table I concerning the commercial grading of coals according to ash content. In addition, a number of editorial improvements have been made in the text.

The following revision is recommended in Specifications D 388:

Table I.—Insert a footnote reference "*a*" to the title of the table and add the following footnote:

> [a] This classification does not include a few coals which have unusual physical and chemical properties and which come within the limits of fixed carbon or B.t.u. of the high-volatile bituminous and subbituminous ranks. All of these coals either contain less than 48 per cent dry, mineral-matter-free fixed carbon or have more than 15,500 moist, mineral-matter-free B.t.u.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 841 (1934); also 1936 Book of A.S.T.M. Tentative Standards, p. 527.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 812 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 520.

[3] These revised specifications were adopted as standard by the Society and appear in the 1937 Supplement to Book of A.S.T.M. Standards, p. 151.—ED.

Both Specifications D 388 and D 389 have been approved as American tentative standards by the American Standards Association (A.S.A. Nos. M20.1 and M20.2-1936, respectively) and are recommended for approval as American standards upon their adoption as A.S.T.M. standards.

The recommendations in the report have been submitted to letter ballot of the committee, which consists of 29 members; 25 members returned their ballots, the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Specifications for Classification of Coals by Grade (D 389 - 34 T), as revised	22	2	1
Specifications for Classification of Coals by Rank (D 388 - 36 T), as revised	23	1	1

ACTIVITIES OF TECHNICAL COMMITTEES

Technical Committee on Coal Classification (H. J. Rose, chairman).—During the past year this technical committee held two meetings; one in Pittsburgh, Pa., on October 20, 1936, and the other in New York City, on February 17, 1937. The Tentative Specifications for Classification of Coals by Rank (D 388 - 36 T) were revised and are being recommended for adoption as standard. The revision, as given earlier in this report, covers the inclusion of a footnote stating that the classification does not apply to certain coals of unusual physical and chemical properties which come within the limits of fixed carbon or B.t.u. of the high-volatile bituminous and subbituminous rank.

The Tentative Specifications for Classification of Coals by Grade (D 389 - 34 T), revised to include size designations, are also being recommended for adoption as standard as appended hereto.[1] A footnote reference in these specifications, concerning the commercial grading of coals according to ash content, is recommended for inclusion in the specifications. This footnote is intended for information and states that ranges in the percentage of ash smaller than 2 per cent as now given in the specifications are commonly used for the commercial grading of coals. The committee believes that the 2 per cent ash increments as given in the specifications are as small as practical for broad general specifications, although ranges in the percentage of ash smaller than 2 per cent are used for the grading of coals for certain commercial purposes.

The Tentative Method for Designating the Size of Coal from Its Screen Analysis (D 431 - 36 T) is recommended for continuation as tentative to afford further opportunity for criticism.

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[1] These revised specifications were adopted as standard by the Society and appear in the 1937 Supplement to Book of A.S.T.M. Standards, p. 151.—ED.

The technical committee continued active work throughout the year, through its subcommittees, as follows:

Subcommittee II on Origin and Composition of Coal and Methods of Analysis (W. A. Selvig, chairman) held a meeting in Pittsburgh, Pa., on October 19, 1936. For several years the subcommittee has considered available information on the microstructure of coals with a view to formulating specifications or definitions for classifying coals according to type, based on origin. The principal factors which determine types of coal are the kinds of vegetation and plant products laid down as ingredient materials, and the environmental conditions controlling their accumulation and their alteration during the peat stage of coal formation. The subcommittee has concluded that the present state of knowledge of types of coal based on origin does not permit formulation of such specifications or definitions at this time. The subcommittee has been relieved of its charge of writing specifications or definitions of types of coal, based on origin.

Subcommittee V on Boundary Lines for Coal Classification (W. A. Selvig, chairman) prepared a footnote, as mentioned above, for the Tentative Specifications for Classification of Coals by Rank (D 388 - 36 T) relative to certain coals of unusual physical properties that are not properly classified according to the specifications. These exempted coals are varieties such as cannels, bogheads and other coals of aquatic origin, which are lower in fixed carbon or higher in B.t.u. than the common banded coals with which they are associated geologically. A study of all available analyses of such coals of the United States and of Canada showed that they could be excluded from the rank classification system by reference to their fixed carbon and B.t.u. content.

Subcommittee VI on Correlation of Scientific Classification with Use Classification (T. W. Harris, Jr., chairman), in cooperation with the National Association of Purchasing Agents, has completed the charts of "Factors Recommended for Consideration in the Selection of Coal" which show the relative importance of chemical and physical factors for the various uses of coal. A report of the subcommittee, including the charts, has been published in booklet form by the National Association of Purchasing Agents, 11 Park Place, New York City, from whom copies may be obtained at a price of $1.00 per copy to cover cost of publication.

Subcommittee VII to Define Coal Sizes and Friability (J. D. Doherty, chairman) held a meeting on October 20, 1936, at Pittsburgh, Pa. The subcommittee recommended the inclusion of size designations in the Tentative Specifications for Classification of Coals by Grade (D 389 - 34 T), such size designations to be in accordance with those given in the Tentative Method for Designating the Size of Coal from Its Screen Analysis

(D 431 - 36 T). The specifications in their revised form as appended hereto[1] include these size designations.

Technical Committee on Nomenclature (E. A. Holbrook, chairman).—During the past year this technical committee held two meetings; one in Pittsburgh, Pa., on December 10, 1936, and the other in New York City, on February 17, 1937. The committee has been assigned the task of formulating definitions of commercially recognized varieties of coal other than those of rank and grade. Satisfactory progress has been made in formulating definitions of varieties such as common banded coal, splint coal, cannel coal, and boghead coal. The committee hopes to present such definitions next year for consideration as tentative.

This report has been submitted to letter ballot of the committee which consist of 29 members; 25 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. FIELDNER,
Chairman.

J. D. BATTLE,
Secretary.

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[1] These revised specifications were adopted as standard by the Society and appear in the 1937 Supplement to Book of A.S.T.M. Standards, p. 151.—ED.

REPORT OF COMMITTEE D-7
ON
TIMBER

Committee D-7 on Timber has carried on its work through its subcommittees, several of which have been very active during the past year, and reports the results herewith.

RECOMMENDATIONS AFFECTING STANDARDS

The recommendations affecting standards are noted in brief form below together with results of the letter ballot. These recommendations are explained in the reports of the subcommittees directly responsible for them.

I. Proposed Tentative Standard.—The committee recommends for publication as tentative the proposed Method of Test for Tar Acids in Creosote and Creosote - Coal Tar Solutions, appended hereto,[1] as proposed by Subcommittee VI on Timber Preservatives.

II. Adoption of Tentative Standards as Standard.—The committee recommends that the following tentative specifications be submitted to letter ballot of the Society for adoption as standard, as proposed by Subcommittee I on Specifications for Timber:

Tentative Specifications for Timber Piles (D 25 – 35 T),[2] and
Tentative Specifications for Structural Wood Joist and Plank, Beams and Stringers, Posts and Timbers (D 245 – 36 T).[3]

The following two typographical errors have been noted in the Tentative Specifications D 245: In Section II of the specifications covering structural beams and stringers, the slope of grain for 1800-lb. *f* structural beams and stringers is indicated as 1 in 16; this should be changed to read "1 in 15." Likewise, in Section III covering structural posts and timbers, the slope of grain for 1100-lb. *c* structural posts and timbers of Douglas fir should be changed from 1 in 12 to read "1 in 10." These corrections will be made when the specifications are next printed.

III. Specifications Continued as Tentative.—The committee recommends that the Tentative Specifications for Zinc Chloride (D 432 – 36 T) be continued as tentative for another year so as to afford ample opportunity for suggestions and criticisms.

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[1] See p. 832.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 868 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 571.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 823 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 547.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 25 members; 23 members returned their ballots, the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. PROPOSED TENTATIVE STANDARD			
Method of Test for Tar Acids in Creosote and Creosote - Coal Tar Solutions	19	0	4
II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Specifications for Timber Piles (D 25 - 35 T)	23	0	0
Specifications for Structural Wood Joist and Plank, Beams and Stringers, and Posts and Timbers (D 245 - 36 T)	23	0	0

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications for Timber (C. E. Paul, chairman).—Subcommittee I had practically completed the revision of its Tentative Specifications for Timber Piles (D 25 - 35 T)[1] last year. Certain criticisms were received which were thoroughly considered during the year and it was finally voted at the meeting of both the subcommittee and Committee D-7 that the specifications as originally presented in 1935 required no further change. As a matter of information, the committee reports that these specifications are in substantial agreement with the specifications developed by the American Railway Engineering Association and by the American Association of State Highway Officials.

The committee voted unanimously to recommend to the Society that, after their adoption as an A.S.T.M. standard, Specifications D 25 be submitted to the American Standards Association for approval as an American standard.

The subcommittee has also continued its studies of the Tentative Specifications for Structural Wood Joist and Plank, Beams and Stringers, and Posts and Timbers (D 245 - 36 T).[2] It has been generally agreed that these specifications as modified last year are now in excellent form and should be adopted as standard. These specifications have already been approved and adopted as standard by the American Railway Engineering Association and will appear in the A.R.E.A. Manual this year. Attention is called to the information concerning these specifications for structural timber appearing in the 1935 and 1936 reports[3] of Subcommittee I.

This subcommittee also reports that it is investigating the possibility of modifying the definition for knots with particular reference to hardwoods so as to clarify methods for measuring knots in both structural timber and piles.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 868 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 571.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 823 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 547.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 362 (1935) and Vol. 36, Part I, p. 443 (1936).

Subcommittee VI on Timber Preservatives (S. R. Church, chairman).—This subcommittee recommends that the Tentative Specifications for Zinc Chloride (D 432 – 36 T) be continued as tentative. No criticism of these specifications has been received and it is intended to recommend their adoption as standard in 1938.

The subcommittee recommends for publication as tentative a proposed Tentative Method of Test for Tar Acids in Creosote and Creosote-Coal Tar Solutions, as appended hereto.[1] This method is identical in substance with the Standard Methods for the Determination of Tar Acids in Creosote Oil of the American Wood-Preservers' Association.

Other subjects which have been given consideration by the subcommittee during the year are the following: (1) proposed revision of the Standard Methods of Test for Insoluble Matter in Creosote (D 367 – 33), on which no action is recommended at present; (2) development of a definition for the term "coal tar" as used in wood preserving, on which no conclusion has as yet been reached; and (3) consideration of the Methods of Sampling and Testing Creosote (D 38 – 33). On the latter, the present methods of sampling are considered satisfactory but may possibly be expanded with more definite instructions for the selection of samples from large tanks and vessels.

The work of the subcommittee during the coming year will include the consideration of unfinished items heretofore noted and the question of the possible adoption of methods for determining the component parts of mixtures of zinc chloride and sodium bichromate as recently reported by the Preservatives Committee of the American Wood-Preservers' Association.

Subcommittee XI on Moisture Content of Timber (M. E. Dunlap, chairman).—Subcommittee XI will continue its work in connection with methods for determining moisture in lumber. Owing to the fact that its chairman was assigned to certain important Governmental duties, the subcommittee regrets that it can only make a progress report this year. The subcommittee has presented certain photographs and descriptions of new moisture methods which are, however, not yet ready for publication.

This report has been submitted to letter ballot of the committee which consists of 25 members; 23 members returned their ballots, of whom 22 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

HERMANN VON SCHRENK,
Chairman.

J. A. NEWLIN,
Secretary.

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[1] See p. 832.

REPORT OF COMMITTEE D-8

ON

BITUMINOUS WATERPROOFING AND ROOFING MATERIALS

Committee D-8 on Bituminous Waterproofing and Roofing Materials has held two meetings during the past year, in Atlantic City, N. J., on July 2, 1936, and in Chicago, Ill., on March 1, 1937.

Recommendations Affecting Standards

I. *Proposed Tentative Standards:*

Proposed Tentative Methods of Test for Sieve Analysis of Mineral Surfacing for Asphalt Roofing and Shingles.—The committee recommends for publication as tentative the following two methods of test for sieve analysis. These methods are a revision of the proposed tests published as information in the 1936 report[1] of Committee D-8, which were based on a cooperative study by Subcommittee XII on Tests for Mineral Surfacing Materials. A further series of cooperative tests has been conducted by the subcommittee as mentioned later in this report.

Proposed Tentative Method of Test for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles,[2] and

Proposed Tentative Method of Test for Sieve Analysis of Non-Granular Mineral Surfacing for Asphalt Roofing and Shingles.[2]

Proposed Tentative Specifications for Asphalt for Damp-proofing and Waterproofing.—Last year the specifications for asphalt for damp-proofing and waterproofing were reverted to a tentative status for the purpose of revising and combining them. This has now been accomplished and the committee has prepared revised specifications for asphalt for damp-proofing and waterproofing, as appended hereto,[3] which are recommended for publication as tentative to replace the following two tentative specifications which are accordingly recommended to be withdrawn:

Tentative Specifications for:

Asphalt for Use in Damp-proofing and Waterproofing Below Ground Level (D 40 – 36 T)[4]

Asphalt for Use in Damp-proofing and Waterproofing Above Ground Level (D 144 – 36 T)[4]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 461, 464 (1936).

[2] See pp. 965, 969.

[3] See p. 927.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 894, 892 (1936); also 1936 Book of A.S.T.M. Tentative Standards, pp. 817, 815.

Proposed Tentative Specifications for Coal-Tar Pitch for Roofing, Damp-proofing and Waterproofing.—These new proposed specifications for coal-tar pitch, as appended hereto,[1] have been prepared to conform to present practice in eliminating differential requirements for type and use. They represent a revision and consolidation of existing specifications for this material. It is recommended that they be accepted for publication as tentative to replace the following six tentative specifications which are accordingly recommended to be withdrawn:

Tentative Specifications for:

High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level (D 42 – 36 T),[2]

High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level (D 145 – 36 T),[2]

High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level (D 200 – 36 T),[2]

High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level (D 201 – 36 T),[2]

High-Carbon Coal-Tar Pitch for Use in Constructing Built-Up Roofs Surfaced with Slag or Gravel (D 251 – 36 T),[2] and

High-Bitumen Coal-Tar Pitch for Use in Constructing Built-Up Roofs Surfaced with Slag or Gravel (D 252 – 36 T).[2]

II. Proposed Revision of Standard:

Standard Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (D 146 – 27).[3]—The following proposed revision of this method specifies a procedure for measuring thickness of specimens to control more adequately the area and force at the contact points of the micrometer. This is highly desirable because there is wide variation in thickness at closely adjacent points and, as felts are compressible, it is preferable to use a dead-weight gage rather than a spring gage. The committee recommends this revision for immediate adoption as standard and accordingly asks for the necessary nine-tenths vote at the annual meeting in order that the revision may be referred to letter ballot of the Society for immediate adoption:

Section 10.—Change from its present form: namely,

10. The thickness of each specimen shall be measured at 10 equally spaced points, 1 in. from a cut edge, with a micrometer gage having flat bearing surfaces at both contact points of not less than $\frac{1}{2}$ in. in diameter and preferably of the spring-and-dial type. The readings for each roll sampled shall be averaged and the minimum, maximum and average thickness calculated to the nearest 0.001 in.

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[1] See p. 948.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 896 to 906 (1936); also 1936 Book of A.S.T.M. Tentative Standards, pp. 834 to 844.

[3] 1936 Book of A.S.T.M. Standards, Part II, p. 1153.

to read as follows:

10. The thickness of each specimen shall be measured at ten equally spaced points at least 1 in. from a cut edge with a dead-weight micrometer gage having a cylindrical foot and anvil with flat bearing surfaces 1 sq. in. in area. The load used shall be 2 lb. per sq. in. The thickness for each roll sampled shall be averaged and the minimum, maximum and average thicknesses calculated to the nearest 0.001 in.

III. Proposed Revision of Tentative Standards:

Tentative Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 - 35 T).[1]—These specifications have been reviewed in connection with several criticisms of the quantity and type of mineral filler in the coating and as a result the committee recommends that they be revised as follows and continued as tentative:

Section 5.—Change requirements (*f*) and (*g*) to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

	65 Pounds		55 Pounds	
	Max., Lb.	Min., Lb.	Max., Lb.	Min., Lb.
(*f*) Weight of *mineral filled* coating [(Soluble in CS_2)] *and surface mineral matter* per 108 sq. ft..........	[20.0]	[15.0] *18.0*	[20.0]	[15.0] *18.0*
(*g*) *Percentage by* weight of mineral matter [per 108 sq. ft.] passing a No. 100 sieve *on basis of total weight of mineral filled coating and surface mineral matter as determined* in (*f*)	[8.0] *50.0 per cent*	[4.0] *15.0 per cent*	[8.0] *50.0 per cent*	[4.0] *15.0 per cent*

New Section.—After the present Section 7 add a new Section 8 on Saturation to read as follows, renumbering the subsequent sections accordingly:

8. *Saturation.*—The felt shall be completely and uniformly saturated, and shall show no unsaturated spots at any point upon cutting strips at random across the entire sheet and splitting them open for their full length.

Tentative Specifications for Asphalt Shingles Surfaced with Coarse Mineral Granules (D 225 - 35 T).[2]—It is recommended that these specifications be revised as follows and continued as tentative:

Section 7.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

7. The felt shall be completely and uniformly saturated, and shall show no unsaturated spots at any point [when the roofing is cut or split open] *upon cutting strips 2 in. in width across the entire shingle and splitting them open for their entire length.*

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 994 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 827.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 998 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 831.

Tentative Specifications for Asphalt Roofing Surfaced with Fine Mineral Granules (D 248 – 35 T).[1]—It is recommended that these specifications be revised by the addition of a new Section 8 identical with that proposed above for inclusion in the Tentative Specifications D 224 – 35 T, renumbering the subsequent sections accordingly.

Tentative Specifications for Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 – 35 T).[1]—It is recommended that these specifications be revised as follows and continued as tentative:

Section 7.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> 7. [Distribution of Coating and Surfacing] *Saturation.*—The felt shall be completely and uniformly saturated, and shall show no unsaturated spots at any point [when the roofing is cut or split open] ***upon cutting strips at random across the entire sheet and splitting them open for their full length.***

Tentative Specifications for Asphalt for Use in Constructing Built-Up Roof Coverings (D 312 – 35 T).[1]—It is recommended that these specifications be revised as follows and continued as tentative:

Section 3.—Change the table of inclines of decks over concrete and requirement (*i*) to read as follows by the addition of the words and figures in italics and the omission of those in brackets:

	Type of Roofing					
	Mineral Surfaced Flat		Unsurfaced Flat		Unsurfaced Steep	
	Min.	Max.	Min.	Max.	Min.	Max.
For use on decks having inclines:						
Over concrete, in. per ft.:						
With nailing	......	3	......	3	3	6
Without nailing	......	***1.5***	......	***1.5***	***1.5***	***3***
(i) Coarse particles retained on No. 200 sieve as percentage of [ash] ***matter insoluble in carbon disulfide,*** per cent	......	[15] ***12***	......	[15] ***12***	......	[15] ***12***

Tentative Specifications for Asphalt Cap Sheet Surfaced with Mineral Granules (D 371 – 33 T).[2]—During the past year these specifications have been reviewed and modified so as to cover both 45-lb. and 55-lb. weights of cap sheet made on No. 33 and No. 50 felts, respectively. The proposed revisions are so extensive as to necessitate a complete rewriting of the specifications. It is accordingly recommended that the specifications be revised as appended hereto,[3] and continued as tentative.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, pp. 990, 986, 983 (1935); also 1936 Book of A.S.T.M. Tentative Standards, pp. 823, 819, 812.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 778 (1933); also 1936 Book of A.S.T.M. Tentative Standards, p. 809.

[3] See p. 924.

Tentative Specifications for Creosote for Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing Below and Above Ground Level (D 43 - 36 T).[1]—In order to bring these specifications into conformity with current requirements it is recommended that they be revised, as appended hereto,[2] and continued as tentative.

Tentative Methods of Testing Asphalt Roll-Roofing Surfaced with Fine Talc, Granular Talc, or Mineral Granules; also Asphalt Shingles Surfaced with Mineral Granules (D 228 - 33 T).[3]—These methods have been carefully reviewed and a cooperative series of tests have been made by six members of Subcommittee IV on Specifications (Including Analytical Methods) for Membrane Materials, to determine what changes are necessary in order to test roofing and shingles conforming to the A.S.T.M. specifications covering these materials, namely, the Tentative Specifications D 224, D 225, D 248, D 249 and D 371. From the results of these tests and from other criticisms received, modifications of the test procedures have been prepared and are incorporated in the completely revised methods appended hereto.[4] As additional changes will probably be made after completion of further studies of the various test procedures, the committee recommends that the methods as revised be continued as tentative.

Tentative Method of Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (D 313 - 35 T).[5]—During the past year, this method has been studied in a series of cooperative tests which indicate the desirability of certain revisions. The changes have been incorporated in the completely revised method appended hereto,[6] which is recommended for publication as tentative to replace the existing method.

At the request of Committee D-15 on Thermometers and Laboratory Glassware, Committee D-8 has approved the inclusion of the following clause as an editorial change in the requirements for high- and low-distillation thermometers:

> For purposes of calibration and certification one or two division markings should be placed below the zero on the Centigrade scale thermometer.

Committee D-8 joins with Committee D-4 on Road and Paving Materials in recommending that Committee E-1 on Methods of Testing give consideration to the following changes in the Tentative Method of Test for Softening Point by Ring-and-Ball Apparatus (E 28 - 36 T):

1. To avoid confusion with present Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (D 36 - 26), change the name of the test to read: "Method of Test for Softening Point by Tapered Ring Apparatus."

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 908 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 846.

[2] See p. 951.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 799 (1933); also 1936 Book of A.S.T.M. Tentative Standards, p. 852.

[4] See p. 957.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, 1001 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 850.

[6] See p. 953.

2. Remove or change the requirement in Section 3 that a sample shall "not be heated more than 30 C. (54 F.) above its softening point, in the case of materials having a softening point below 121 C. (250 F.) . . . or more than 50 C. (90 F.) for . . . materials with softening points above 121 C. (250 F.)."

The change in Section 3 is suggested since certain asphaltic materials require greater differences than these between the softening point and pouring temperature.

The committee further recommends that wide publicity be given to the fact that Method E 28 – 36 T is essentially a new method which gives somewhat different results from those obtained by the Standard Method D 36 – 26. Committee D-8 does not favor the withdrawal of Standard Method D 36 – 26 nor the adoption as standard of Method E 28 – 36 T until these recommendations are adopted and Method E 28 – 36 T is acceptable to the members of the committee.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. PROPOSED TENTATIVE STANDARDS			
Method of Test for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles	32	1	4
Method of Test for Sieve Analysis of Non-Granular Mineral Surfacing for Asphalt Roofing and Shingles	32	1	4
Specifications for Asphalt for Damp-proofing and Waterproofing, to replace Specifications D 40 – 36 T and D 144 – 36 T	35	1	1
Specifications for Coal-Tar Pitch for Roofing, Damp-proofing and Waterproofing, to replace Specifications D 42 – 36 T, D 145 – 36 T, D 200 – 36 T, D 201 – 36 T, D 251 – 36 T and D 252 – 36 T	32	3	2
II. PROPOSED REVISION OF STANDARD			
Standard Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (D 146 – 27), immediate adoption	33	0	2
III. PROPOSED REVISION OF TENTATIVE STANDARDS			
Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 – 35 T)	32	2	3
Specifications for Asphalt Shingles Surfaced with Coarse Mineral Granules (D 225 – 35 T)	33	1	3
Specifications for Asphalt Roofing Surfaced with Fine Mineral Granules (D 248 – 35 T)	33	1	3
Specifications for Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 – 35 T)	33	1	3
Specifications for Asphalt for Use in Constructing Built-Up Roof Coverings (D 312 – 35 T)	32	1	4
Specifications for Asphalt Cap Sheet Surfaced with Mineral Granules (D 371 – 33 T)	31	1	5
Specifications for Creosote for Priming Coat with Coal-Tar Pitch in Dampproofing and Waterproofing Below and Above Ground Level (D 43 – 36 T)	32	0	4
Methods of Testing Asphalt Roll Roofing Surfaced with Fine Talc, Granular Talc or Mineral Granules; also Asphalt Shingles Surfaced with Mineral Granules (D 228 – 33 T)	33	0	4
Method of Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (D 313 – 35 T)	34	0	3

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 58 voting members; 38 members returned their ballots, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee IV on Specifications (Including Analytical Methods) for Membrane Materials (E. H. Berger, chairman).—During the past year this subcommittee has carefully reviewed all of the standards and tentative standards for which it had been primarily responsible and as a result of its study, has recommended numerous revisions which have been incorporated in this report.

Subcommittee V on Specifications for Bituminous Coatings for Cold Application (E. O. Rhodes, chairman).—This subcommittee is responsible for the proposed revision of the Tentative Specifications for Creosote for Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing Below and Above Ground Level (D 43 – 36 T), mentioned above. During the coming year, it expects to offer revisions of the companion specifications for asphalt primer (D 41 – 36 T). The subcommittee is also making a special study of methods of determining the consistency of various bituminous coatings for cold application.

Subcommittee VIII on Accelerated Weathering Tests (O. G. Strieter, chairman).—This subcommittee is continuing its study of the Proposed Method for Accelerated Weathering Tests on Bituminous Materials[1] but has no definite recommendations to make at this time. However, it is believed that a description of the cycle now generally used in this test should be published for information. The proposed change in the weathering cycle consists in a more effective use of water, which is a very destructive agent in the weathering process. The subcommittee is now gaining experience with this modified cycle and hopes to make definite recommendations at a later date. The proposed cycle is as follows:

Test Procedure.—The test specimens shall be subjected to a daily weathering cycle consisting of periods of exposure to cold, light, and water, the weathering cycle being as follows:

Rain exposure for a period of ¼ hr.
Cold exposure for a period of 1¾ hr.
Light-spray for a period of 4 hr.
Rain exposure for a period of ½ hr.
Light exposure for a period of 17 hr.

For the light, rain, and light-spray exposure period, the specimens shall be placed in the holders of the cylinder. For the rain period the lamp shall be raised above the cylinder, the latter covered, and the specimens vigorously sprinkled with water by means of a rotary spray which operates at the bottom of the cylinder.

For the cold-exposure period, the specimens shall be placed in the refrigerator, previously cooled to −10 F. (−23 C.). Fifteen minutes are allowed to change panels from the cylinder to the refrigerator and the same time is again allowed after the cold period to return the specimens to the cylinder for the light-spray period.

For the light period the lamp shall be operated with the revolving cylinder. During the light-spray period, the light from the lamp shines on the specimens continuously while the water, running constantly from the spray at a fixed point, wets the specimens in the revolving cylinder once every 20 min.

The temperature at the surfaces of the specimens during the light period shall be 135 to 145 F. (57 to 63 C.). In the case of the light-spray period the specimens will, of course, be cooled as they pass by the water spray; the specimens shall, however, be allowed to reach a temperature (warm) of about 140 F. (60 C.) before being wet again. (In cold weather, it may be necessary to use warm water for spraying in order to reach such a temperature). Water (2 to 4 in. in depth) shall be kept in the tank or sump all the time, the excess water flowing out over the overflow.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 381 (1933).

Subcommittee IX on Bituminous Emulsions (Lester Kirschbraun, chairman).—This subcommittee has for some time been engaged in formulating tests for emulsions to cover two sets of characteristics, as follows: (1) the resistance to water action, and (2) the ability to withstand flow at elevated temperatures. Both methods involved the plating out of the emulsion under definite conditions and in quantities such as to produce dry residues of predetermined film thickness.

After obtaining dried films on the selected surface, the emulsions were tested for resistance to water action by immersion in water for 24 hr., followed by physical examination of the film. A similarly prepared dried film which had been plated on steel was subjected to a temperature of 176 F. (80 C.) for 2 hr. in accordance with the test for behavior on heating as described in Section 17 of the Tentative Methods of Testing Asphalt Roll-Roofing Surfaced with Fine Talc, Granular Talc or Mineral Granules; also Asphalt Shingles Surfaced with Mineral Granules (D 228 – 33 T),[1] and observed for flow of the emulsion.

TABLE II.—RESULTS OF COOPERATIVE TESTS ON BITUMINOUS EMULSIONS.

Test	Bituminous Emulsion Sample	Observations Reported by Cooperating Laboratories					
		Headley Asphalt Co.	N. Y. State Dept. of Public Works	Texas Co.	Barrett Co.	The Barber Co., Inc.	Flintkote Co.
Resistance to Flow Under Heat After 2-hr. Exposure at 176 F. (80 C.), Emulsion on Steel Plates	No. 1	Severe	Flows	Flows	Flows	Flows	Flows
	No. 2	None	Flows	Flows[a]	Flows	Flows	Flows
	No. 3	None	None	None	None	None	None
RESISTANCE TO WATER ACTION AFTER 24-HR. SUBMERSION							
Emulsion on Steel Plates:							
Adhesion	No. 1	Fair	Strips	Poor	Strips	Very poor	Strips
	No. 2	Fair	Strips	Poor	Strips	Very poor	Strips
	No. 3	Good	Good	Good	Good	Good	Good
Cohesion	No. 1	......	Good	Good	Good	Good	Good
	No. 2	......	Good	Good	Good	Fair	Good
	No. 3	......	Good	Good	Good	Good	Good
Re-emulsifies	No. 1	No	No	No	No	Slight	None[b]
	No. 2	No	No	No	No	No	None
	No. 3	No	No	No	No	No	None
Emulsion on Tile Base:							
Adhesion	No. 1	Good	Good	Good	Good	Good	Strips slightly
	No. 2	Good	Fair	Good	Good	Very poor	Good
	No. 3	Fair	Good	Good	No Test[c]	Good	Good
Cohesion	No. 1	......	Good	Good	Good	Good	Good
	No. 2	......	Good	Good	Good	Fair	Good
	No. 3	......	Good	Good	No Test	Good	Good
Re-emulsifies	No. 1	No	No	No	No	No	No
	No. 2	No	No	No	No	No	No
	No. 3	No	No	No	No Test	No	No

[a] When "thoroughly dry," slight sagging.
[b] No re-emulsification but needle revealed unbroken emulsion underneath which re-emulsified on further soaking.
[c] Emulsion cracked.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 799 (1933); also 1936 Book of A.S.T.M. Tentative Standards, p. 852.

Table III.—Results of Cooperative Test Studies of the Proposed Method of Test for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles.

Cooperating Laboratories		Retained on U. S. Standard Sieve						Passing Sieve No. 70
		No. 12	No. 16	No. 20	No. 30	No. 40	No. 70	
Green Ceramic Roofing Granules								
Minnesota Mining and Mfg. Co.	First Analysis	0.2	29.4	44.2	20.0	5.4	0.9	0.05
	Check Analysis	0.1	29.2	44.1	20.2	5.4	1.1	0.14
Central Commercial Co.	Analysis	0.36	29.42	41.92	22.95	4.09	1.03	0.23
Minnesota Mining and Mfg. Co.	Second Analysis	0.2	29.7	44.0	20.0	4.9	1.1	....
Minnesota Mining and Mfg. Co.	First Analysis	0.2	28.9	44.4	20.6	5.1	0.8	0.06
	Check Analysis	0.2	28.9	44.2	20.9	5.0	0.8	0.13
Barrett Co.	Analysis	0.1	27.3	41.3	25.1	5.0	1.0	0.2
Minnesota Mining and Mfg. Co.	Second Analysis	0.1	29.1	44.0	20.6	4.8	1.2	0.3
Minnesota Mining and Mfg. Co.	First Analysis	0.2	29.5	43.3	20.8	5.2	1.0	0.04
	Check Analysis	0.1	29.3	43.2	21.0	5.3	1.1	0.15
R. J. Funkhouser and Co.	Analysis	0.14	29.08	44.9	19.96	4.84	1.13	0.08
Minnesota Mining and Mfg. Co.	Second Analysis	0.2	29.5	43.9	20.0	5.1	1.2	....
	Check Analysis	0.2	29.3	44.1	20.1	5.2	1.1	....
Minnesota Mining and Mfg. Co.	First Analysis	0.2	28.4	44.0	20.9	5.2	1.0	0.01
	Check Analysis	0.2	28.2	44.3	20.9	5.3	1.1	0.91
Bird and Son, Inc.	First Analysis	0.08	27.13	39.67	25.34	6.55	0.91	0.19
	Second Analysis	0.04	27.19	39.06	24.93	7.27	1.12	0.30
Minnesota Mining and Mfg. Co.	Second Analysis	0.2	27.8	44.2	21.5	5.3	1.1	....
	Check Analysis	0.2	28.5	44.2	20.8	5.2	1.1	....
Minnesota Mining and Mfg. Co.	First Analysis	0.2	29.6	44.0	20.3	5.0	0.9	0.07
	Check Analysis	0.2	29.4	44.0	20.4	5.1	0.9	0.14
Philip Carey Mfg. Co.	First Analysis	0.5	29.8	40.9	23.3	4.6	0.8	0.1
	Second Analysis	0.4	30.9	41.0	22.4	4.3	0.9	0.1
	Third Analysis	0.5	30.7	40.8	22.6	4.4	0.9	0.2
Minnesota Mining and Mfg. Co.	Second Analysis	0.2	29.4	44.4	20.3	4.7	0.9	....
	Check Analysis	0.2	29.1	44.2	20.5	5.0	1.0	....
Minnesota Mining and Mfg. Co.	First Analysis	0.2	28.9	44.9	20.9	5.1	0.8	0.06
	Check Analysis	0.1	29.0	44.8	21.0	5.1	0.1	0.13
The Barber Co., Inc.	First Analysis	0.1	26.0	43.9	24.9	4.1	0.9	0.1
	Second Analysis	0.1	25.4	44.0	24.9	4.6	0.9	0.1
Green Slate Roofing Granules								
Minnesota Mining and Mfg. Co.	First Analysis	1.0	26.8	39.1	23.6	8.4	1.1	0.13
	Check Analysis	0.9	26.7	39.0	23.7	8.6	1.0	0.18
Central Commercial Co.	Analysis	1.26	25.03	36.99	26.58	8.8	1.11	0.23
Minnesota Mining and Mfg. Co.	Second Analysis	1.0	25.6	39.4	23.9	8.9	1.1	0.3
Minnesota Mining and Mfg. Co.	First Analysis	1.0	25.4	38.8	24.2	9.3	1.1	0.2
	Check Analysis	0.9	25.2	38.9	24.1	9.5	1.2	0.23
Barrett Co.	Analysis	0.7	24.5	36.9	27.8	8.7	1.2	0.2
Minnesota Mining and Mfg. Co.	Second Analysis	1.0	24.5	39.4	24.3	9.1	1.2	0.4
Minnesota Mining and Mfg. Co.	First Analysis	0.9	25.7	39.6	23.9	8.7	1.2	0.13
	Check Analysis	0.9	25.7	39.5	23.9	8.9	1.2	0.16
R. J. Funkhouser and Co.	Analysis	1.1	24.6	40.7	23.6	8.4	1.5	0.2
Minnesota Mining and Mfg. Co.	Second Analysis	1.0	25.2	39.2	24.3	9.1	1.2	0.4
Minnesota Mining and Mfg. Co.	First Analysis	0.8	25.3	39.9	24.0	9.0	1.1	0.02
	Check Analysis	0.7	25.0	39.6	24.2	9.2	1.1	0.2
Bird and Son, Inc.	First Analysis	0.43	24.31	36.21	27.05	10.84	1.08	0.16
	Second Analysis	0.43	23.95	35.60	27.59	10.83	1.12	0.24
Minnesota Mining and Mfg. Co.	First Analysis	0.9	24.5	39.4	24.7	9.4	1.2	0.17
	Check Analysis	0.9	24.4	39.3	24.9	9.4	1.3	0.2
Johns-Manville Corp.	Analysis	0.9	24.5	34.6	29.7	8.6	1.5	0.2
Minnesota Mining and Mfg. Co.	First Analysis	0.9	24.6	39.4	24.7	9.0	1.3	0.12
	Check Analysis	0.7	24.7	39.6	24.5	9.1	1.2	0.18
Philip Carey Mfg. Co.	First Analysis	1.2	23.1	37.0	28.1	9.3	1.1	0.2
	Second Analysis	1.1	22.7	36.8	28.9	9.2	1.1	0.2
	Third Analysis	1.1	22.8	36.9	28.9	9.2	1.0	0.2
Minnesota Mining and Mfg. Co.	Second Analysis	0.8	23.7	39.9	24.8	9.4	1.4	....
	Check Analysis	0.9	23.9	40.1	24.5	9.2	1.4	....
Minnesota Mining and Mfg. Co.	First Analysis	0.9	24.7	39.6	24.6	9.0	1.2	0.09
	Second Analysis	0.8	24.6	39.5	24.8	9.0	1.1	0.19
The Barber Co., Inc.	First Analysis	0.4	20.1	38.9	30.4	9.1	1.0	0.1
	Second Analysis	0.7	21.0	38.7	28.9	9.1	1.7	0.7

On account of the variety of surfaces to which emulsions might be applied in service, it was desired to select a plating surface other than steel which might afford a suitable base for test applications in the laboratory. A standard grade of unfired ceramic tile was obtained for this purpose and was used in these cooperative tests.

TABLE IV.—RESULTS OF COOPERATIVE TEST STUDIES OF THE PROPOSED METHOD OF TEST FOR SIEVE ANALYSIS OF NON-GRANULAR MINERAL SURFACING FOR ASPHALT ROOFING AND SHINGLES.

Cooperating Laboratories		Retained on U. S. Standard Sieve									Passing Sieve No. 100	Passing Sieve No. 200	Loss and Error	Sieving Time, min.
		No 20	No. 30	No. 40	No. 50	No. 60	No. 70	No. 80	No. 100	No. 200				
						COARSE MICA								
The Barber Co., Inc.	First Analysis	4.5	24.5	23.8	23.2	....	12.5	....	6.7	....	4.8	....	....	30
	Check Analysis	5.2	24.0	23.6	23.0	....	12.7	....	6.5	....	5.0	....	....	30
Barrett Co.	Analysis	6.5	....	48.7	23.1	....	11.2	....	6.2	....	4.3	....	0.5	30
Bird and Son, Inc.	First Analysis	6.3	29.0	24.6	20.5	....	10.4	....	5.2	....	4.0	....	....	30
	Check Analysis	7.1	29.3	22.7	21.2	....	10.5	....	4.5	....	4.7	....	....	30
Philip Carey Mfg. Co.	First Analysis	4.6	25.4	27.7	19.7	....	11.6	....	6.1	....	4.9	....	0.6	30
	Second Analysis	4.7	25.2	27.3	20.4	....	11.8	....	5.9	....	4.7	....	0.7	30
	Third Analysis	4.8	25.3	27.4	20.1	....	11.6	....	6.0	....	4.8	....	0.5	30
R. J. Funkhouser and Co.	Analysis (Wayne)	5.5	20.5	27.6	23.0	8.0	....	8.3	2.5	....	4.6	....	0.2	60
	Analysis (Delta)	4.0	19.3	29.5	22.2	4.4	....	....	15.2	....	5.4	....	3.0	60
						FINE MICA								
The Barber Co., Inc.	First Analysis	....	0.8	0.6	6.4	....	14.0	....	16.5	24.7	...	37.0	...	60
	Check Analysis	....	0.7	0.7	6.0	....	13.8	....	16.6	25.4	...	36.8	...	60
Barrett Co.	Analysis	....	0.3	0.4	6.8	....	14.4	....	16.2	26.7	...	35.2	3.1	70
Bird and Son, Inc.	First Analysis	....	0.3	0.2	8.0	....	16.6	....	17.1	33.5	...	24.3	...	60
	Check Analysis	....	0.4	0.4	7.6	....	16.7	....	16.6	32.5	...	25.8	...	60
Philip Carey Mfg. Co.	First Analysis	....	0.3	0.5	5.6	....	15.9	....	18.2	26.8	...	32.7	0.3	60
	Second Analysis	....	0.3	0.6	5.7	....	15.1	....	18.3	27.2	...	32.8	0.9	60
	Third Analysis	....	0.3	0.6	5.9	....	14.9	....	18.3	27.8	...	32.5	0.5	60
R. J. Funkhouser and Co.	Analysis (Wayne)	....	0.2	0.1	5.5	8.4	....	19.4	10.0	22.6	...	33.8	0.7	105
	Analysis (Delta)	....	0.6	0.6	7.5	8.5	....	....	22.4	26.1	...	34.3	0.6	60
						TALC FLOUR								
The Barber Co., Inc.	First Analysis	....	....	....	0.0	....	0.1	....	0.3	2.6	...	97.0	...	30
	Check Analysis	....	....	....	0.0	....	0.1	....	0.2	2.5	...	97.2	...	30
Barrett Co.	Analysis	....	0.0	0.0	0.0	....	0.0	....	0.1	5.1	...	94.8	1.4	40
Bird and Son, Inc.	First Analysis	....	....	....	0.0	....	0.0	....	0.2	7.4	...	92.4	...	100
	Check Analysis	....	....	....	0.0	....	0.0	....	0.2	6.4	...	93.4	...	100
Philip Carey Mfg. Co.	First Analysis	....	0.0	0.0	0.0		0.1	....	0.2	3.8	...	96.0	1.1	40
	Second Analysis	....	0.0	0.0	0.0	....	0.1	....	0.3	4.0	...	95.6	1.0	40
	Third Analysis	....	0.0	0.0	0.0	....	0.1	....	0.2	4.4	...	95.3	0.7	40
R. J. Funkhouser and Co.	Analysis (Wayne)	....	0.0	0.0	0.0	0.1	....	0.1	0.1	4.8	...	94.9	0.1	90
	Analysis (Delta)	....	0.0	0.0	0.0	0.0	....	....	0.0	4.5	...	95.5	1.0	90

The observations reported by the cooperating laboratories, as given in Table II, appear to be reasonably concordant, and it is believed that after some further discussion and consideration the subcommittee will be able to formulate a final report. At a meeting on June 28, Committee D-8 ordered to letter ballot, a new proposed Tentative Method of Testing Films Deposited from Bituminous Emulsions which, if approved, will be submitted to Committee E-10 on Standards for publication as tentative by the Society.[1]

Subcommittee XII on Tests for Mineral Surfacing Materials (H. W. Greider, chairman).—In connection with the two proposed Tentative Methods of Test for Sieve Analysis of Granular and Non-granular Mineral Surfacing for Asphalt Roofing and Shingles, mentioned earlier in this report, Subcommittee XII has conducted a series of cooperative tests, the results of which are presented in the accompanying Tables III and IV.

This report has been submitted to letter ballot of the committee which consists of 58 voting members; 38 members returned their ballots, of whom 37 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

J. M. WEISS,
Chairman.

PRÉVOST HUBBARD,
Secretary.

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[1] See Editorial Note below.

EDITORIAL NOTE

Subsequent to the annual meeting Committee D-8 on Bituminous Waterproofing and Roofing Materials presented to the Society through Committee E-10 on Standards proposed Tentative Methods of Testing Films Deposited from Bituminous Emulsions. These methods were accepted by Committee E-10 on August 26, 1937, and appear on p. 955.

REPORT OF COMMITTEE D-9

ON

ELECTRICAL INSULATING MATERIALS

Committee D-9 on Electrical Insulating Materials held two meetings during the year and will hold a third during the annual meeting of the Society in June at New York, N. Y. The meetings were held in October, 1936, and February, 1937, with an average attendance of 52 members and 30 visitors. At these meetings considerable progress was made in the work which has been pending before the committee and several projects have reached the stage where definite recommendations were made resulting in additions or changes in the standards for which Committee D-9 is responsible. There are also a number of other projects still under way which are mentioned briefly later in this report.

Recommendations Affecting Standards

As a result of the year's work, the committee is submitting tentative revisions of two existing standards and is recommending revisions of seven tentative standards. The adoption of a tentative revision in one standard is also recommended. The recommendations being presented are included in detail in Appendix I, each being accompanied by a brief explanation of the reason for the proposed revision together with data indicating the scope of the investigations substantiating the recommendations. The standards affected by these revisions are listed in the following table.

These recommendations have been submitted to letter ballot of the committee which consists of 78 voting members; 52 members returned their ballots, with the results shown in the following table:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. Proposed Tentative Revisions of Standards			
Methods of Testing Electrical Porcelain (D 116 - 34)	27	0	25
Methods of Test for Resistivity of Insulating Materials (D 257 - 33)	41	0	11
II. Proposed Revisions of Tentative Standards			
Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 - 36 T)	27	0	25
Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 36 T)	30	0	22
Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 - 36 T)	32	0	20
Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 - 35 T)	25	0	27
Methods of Testing Shellac Used for Electrical Insulation (D 411 - 35 T)	22	0	30
Specifications for Flexible Varnished Tubing Used in Electrical Insulation (D 372 - 35 T)	22	0	30
Specifications for Black Bias-Cut Varnished Cloth Tape Used for Electrical Insulation (D 373 - 36 T)	25	0	27
III. Adoption of Tentative Revision of Standard as Standard			
Methods of Testing Molded Materials Used for Electrical Insulation (D 48 - 33)	32	0	20

ACTIVITIES OF SUBCOMMITTEES

A large part of the work of the subcommittees during the year has consisted in obtaining data and studying results with a view to improving the present standards and developing new standards. In any one year only a small portion of the work can result in definite recommendations. It is felt desirable, therefore, to mention briefly some of the other projects which have engaged the attention of the subcommittees, but which as yet have not reached the stage where definite standards have resulted.

A new subcommittee on conditioning has been organized, which brings the total number of subcommittees to ten.

Subcommittee I on Insulating Varnishes, Paints and Lacquers (C. F. Hanson, chairman).—The work on saturant varnish is still active and experimental studies will be continued in four laboratories. The work on specifying a standard procedure for sampling varnish has been abandoned because of lack of interest. The work on definitions for various types of varnishes is still in progress.

Subcommittee II on Molded Insulating Materials (W. A. Evans, chairman).—The work on shrinkage and standard molds is still in progress. The work during the past year indicated that the type of mold affected the results. There are now available 800 test bars which will be tested in an effort to establish a standard mold. Work on cure tests, arc-resistance test and conditioning has also been in progress.

Subcommittee III on Plates, Tubes and Rods (Dean Harvey, chairman). —In order to determine the necessity for the conditioning of samples before testing, a set of 2400 samples of sheet materials has been prepared. They are being tested for impact strength, Rockwell hardness, and power factor after exposure to different degrees of conditioning. Extensive tests of sheet materials by the proposed test for arc resistance in four laboratories did not give sufficiently consistent results to justify approval and a further study is being made.

The acetone test for determining the degree of cure of laminated phenolic products has been outlined and is being given a trial in several laboratories.

A new compound die has been designed for punching sheet material and punching quality will be compared with Rockwell *B* hardness on heated specimens to determine whether the Rockwell test will indicate punching quality.

Methods of test for identity of phenolic materials are being prepared at the request of the U. S. Navy Department.

A method for the measurement of the thickness of large sheets is being prepared at the request of the National Electrical Manufacturers' Association.

In response to an inquiry for a method of test for deterioration due to heat, it was decided that a single test for this purpose is not practicable. The effect of heat should be determined upon the particular property in question.

Specifications for laminated phenolic materials for radio use have been prepared at the request of the Radio Manufacturers' Association and are being submitted to the industry for comments.[1]

At the request of the National Electrical Manufacturers' Association consideration will be given to the development of methods of test for vulcanized fiber. Methods for determining water absorption and Brinell hardness have been outlined and considerable test work has been completed but some changes in the methods are necessary.

Subcommittee IV on Insulating Mineral Oils (E. A. Snyder, chairman).—Subcommittee IV has prepared a revision of the Proposed Method of Test for Neutralization Number of Petroleum Products and Lubricants (Potentiometric Method) published as information last year.[2] The revised method is being studied by members of the subcommittee but is not ready for presentation as tentative.

The work on life test of oils has been outlined to include studies of oxidation tests in the presence of catalysts. The program is rather lengthy and will require considerable time before a final report can be made. The work on power factor and dielectric constant has been somewhat delayed due to the fact that the test cell being purchased through the Research Fund of the Society has not been delivered.

Other work in progress includes methods for gas content of oil, power factor of treated paper and a study of the dielectric strength test procedure. The subcommittee has also been active in translating and abstracting papers received from the International Electrochemical Commission and also comments and discussions received from abroad on the paper presented at the 1936 annual meeting entitled, "Testing for Sludge Formation in Mineral Transformer Oil."[3]

Subcommittee V on Ceramic Products (*Porcelain, Glass, etc.*) (L. E. Barringer, chairman).—Subcommittee V has been unusually active during the past year. Attention was given to the criticisms of the Standard Methods of Testing Electrical Porcelain (D 116 – 34) received when the methods were presented to the American Standards Association. It is believed that these items have been cleared up in the revision being recommended as tentative as given in Appendix I. The subcommittee has also considered methods for testing titania bodies having high dielectric constant and low power factor, and also steatitic bodies. For the present it is felt that the test procedures covered in Methods D 116 will apply.

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[1] Subsequent to the annual meeting Committee D-9 presented these specifications as tentative to the Society through Committee E-10 on Standards, see Editorial Note, p. 429.—ED.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 409 (1936).

[3] F. M. Clark and E. A. Snyder, "Testing for Sludge Formation in Mineral Transformer Oil," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 568 (1936).

The Section on Glass presented a report at the meeting in February, 1937. Due to the establishment by the Society of a standing committee on glass, this section arranged to cooperate and will continue as the unit for developing test methods for glass when used as electrical insulation.[1]

Subcommittee VI on Solid Filling and Treating Compound (M. F. Skinker, chairman).—Subcommittee VI investigated the dielectric strength procedure for filling and treating compounds as a result of some criticism of the method. Slight changes in the test have been completed and the revised procedure is being recommended for inclusion in the Tentative Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 – 36 T), as given in Appendix I.

Subcommittee VII on Electrical Tests (J. A. Scott, chairman).—Last year this subcommittee presented a completely revised method for measurement of power factor and dielectric constant. This year it has presented a complete revision, appended hereto,[2] of the Standard Methods of Test for Resistivity of Insulating Materials (D 257 – 33) for publication as tentative as explained in Appendix I.

The oil cell being purchased through the Research Fund of the Society was not delivered in time to circulate it in the subcommittee for round-robin tests. The work on power factor of solid insulation is in progress and five laboratories have completed their measurements. There is now under way a complete revision of the method of test for dielectric strength. This work will include all of the test procedures now described in the standard methods under the jurisdiction of Committee D-9.

Subcommittee VIII on Insulating Papers and Fabrics (Treated and Untreated) (R. W. Chadbourn, chairman).—The Section on Paper is continuing its examination of various methods of acid extraction from insulating papers. A new method, involving extraction by cold water, is now being investigated. In the study of oil penetration, the effects of many variables have been determined and a comparative examination has been made of several types of test apparatus. The section is also reviewing the procedure for determining tensile strength described in the A.S.T.M. Tentative Methods of Testing Untreated Paper Used in Electrical Insulation (D 202 – 36 T) and the Technical Association of the Pulp and Paper Industry's method for tensile strength with a view not only to improving and bringing up to date the A.S.T.M. method but also to bringing the two methods into closer agreement, if possible.

The Section on Thickness is studying the use of the dial type and also an improved machinist's type of micrometer. In this improved type, the pressure on the test specimen can be accurately controlled.

The Section on Fabrics is continuing to accumulate data on the effect of humidity and elongation on the dielectric strength of varnished cloth tapes. Joint consideration of the step-by-step dielectric strength test method by groups representing Subcommittee VIII and the Insulated Power Cable Engineers' Association has been arranged with a view to eliminating present differences in test standards.

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[1] Subsequent to the annual meeting Committee D-9 presented these methods as tentative to the Society through Committee E-10 on Standards, see Editorial Note, p. 429.—ED.

[2] See p. 1016.

Subcommittee IX on Mica Products (M. P. Davis, chairman).—Subcommittee IX has the samples of natural mica which are to be used for power-factor tests ready for distribution. The problem of electrode preparation has presented some difficulties. Attempts are being made to prepare color photographs as an aid in the classifications of mica. It is also believed that ionization tests may assist in the classification.

The work on pasted mica has in general dealt with tests on binder contents and tests for stability under heat and pressure.

Subcommittee X on Conditioning (Robert Burns, chairman).—This subcommittee, which has functioned formerly as a special subcommittee, now becomes one of the regular subcommittees. It will continue to serve the other subcommittees in supplying information on conditioning methods. It also has under consideration the problem of specifying a normal room condition which can be economically obtained. The subcommittee is also investigating oven equipment for air-drying and baking varnishes.

Special Subcommittee on Significance of Tests (Dean Harvey, chairman). —In the development of suitable statements concerning the significance of various tests included in the standards sponsored by Committee D-9, it was felt desirable to publish several of the proposals as information only in this report and they accordingly appear in Appendix II. These proposals are being submitted for comment and criticism after which a general type of statement can be developed. Ultimately, it is hoped to have statements covering most of the methods of test for electrical insulating materials, which should enable the users readily to appreciate the purpose and limitations of the various tests.

This report has been submitted to letter ballot of the committee, which consists of 78 voting members; 52 members returned their ballots, of whom 49 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

T. SMITH TAYLOR,
Chairman.

E. J. RUTAN,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-9 on Electrical Insulating Materials presented to the Society on August 26, 1937, through Committee E-10 on Standards the following recommendations:

Proposed Tentative Specifications for Phenolic Laminated Sheet for Radio Applications,

Proposed Tentative Methods of Testing Pin-Type, Lime Glass Insulators, and

Proposed revision of Tentative Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 37 T).

The above recommendations were accepted by Committee E-10 and appear on pp. 973; 1008, and 431, respectively.

APPENDIX I

PROPOSED REVISIONS IN STANDARDS FOR ELECTRICAL INSULATING MATERIALS

In this Appendix are given proposed revisions in certain standard and tentative standards covering electrical insulating materials which are referred to earlier in this report. In connection with each title is given the reference to the publication in which the standard appears in its present form.

Proposed Tentative Revisions of Standards

Tentative Revision of Standard Methods of Testing Electrical Porcelain (A.S.T.M. Designation: D 116 – 34):[1]

During the consideration of these methods by the American Standards Association for approval as American standard, some criticism was advanced in regard to the scope. Accordingly, the following change in these standard methods is recommended for publication as a tentative revision:

Section 1.—Change this section and the Note from their present form: namely,

> 1. These methods are intended to apply to porcelain and other vitrified ceramic materials used in the manufacture of electrical insulating parts.
>
> Note.—Tests on porcelain are made on unglazed specimens since it is desired to determine the strength of the porcelain structure only. Tests have indicated, however, that the glaze may have an effect on the mechanical strength. In some cases the values obtained are higher and in other cases lower than obtained on unglazed specimens.

to read as follows:

> 1. These methods outline procedures for making the various tests described when they are required for an investigation or examination of porcelain or similar vitrified ceramic materials used as electrical insulating mediums. They are not to be interpreted as forming test specifications for complete insulators.
>
> Note.—The glaze on a porcelain object may affect certain properties of the material, depending not only upon its presence but also upon its thickness and composition. While the test methods have been written with the intent of applying to unglazed specimens, they are equally suitable for glazed specimens. The report of test should in all cases indicate whether the specimens are glazed or unglazed.

Tentative Revision of Standard Methods of Test for Resistivity of Insulating Materials (A.S.T.M. Designation: D 257 – 33):[2]

A revision of these methods, based on comments received during past years, has been prepared in which the subject matter has been considerably shortened and simplified. The use of shielding to drain off leakage currents has been included. The condenser method suitable for higher ranges of resistance of less interest in routine work has been omitted. The changes recommended have been incorporated in the completely revised proposed Tentative Methods of Test for Insulation Resistance of Electrical Insulating Materials, appended hereto,[1] which the committee recommends for publication as a tentative revision and which are intended to replace when adopted the present Standard Methods D 257.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1178.

[2] *Ibid.*, p. 1203.

Proposed Revisions of Tentative Standards

Revision of Tentative Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (A.S.T.M. Designation: D 176 – 36 T):[2]

As a result of some recent criticism of the section on dielectric strength, a study was made of the present method and two new methods. It was found that the new methods had no advantages to offer if the old method was revised to provide for a slightly longer container. Accordingly, a completely revised dielectric strength test, covering Sections 33 to 38, inclusive, and Appendix III of the methods, is appended hereto[3] and is recommended for inclusion in Methods D 176. This proposed revision answers the criticism advanced on the present method.

Revision of Tentative Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229 – 36 T):[4,5]

At the request of the National Electrical Manufacturers' Association, methods of test have been prepared for density, incombustible material determined by the ash test, zinc chloride and silica. These methods correspond to the procedures which have been in use for these tests.

A test for flammability has been developed at the request of the U. S. Navy Department. The method of test for flammability has been tried in three laboratories on grades X, XX and C of laminated phenolic sheet material, commercial fiber and bone fiber with the following results:

Grade	Time Required for Ignition (Average of Five Tests), Sec.		
	Laboratory 1	Laboratory 2	Laboratory 3
X	92	106	95
XX	117	121	112
C	91	93	92
Commercial fiber	23	30	36
Bone fiber	39	49	44

Three samples of laminated phenolic sheets $\frac{1}{8}$ in. in thickness with different volatile contents were tested and the volatile content within the ranges supplied had little effect on the inflammability as indicated by the following data:

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[1] See p. 1016.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1000 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 871.

[3] These changes have been incorporated in the revised tentative methods, see pp. 997 and 1005.—Ed.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 990 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 986.

[5] Subsequent to the annual meeting additional revisions of these methods were presented to Committee E-10 on Standards, see Editorial Note, p. 429. The revisions have been incorporated in the revised tentative methods, see p. 1027.—Ed.

VOLATILE CONTENT, PER CENT	TIME REQUIRED FOR IGNITION (AVERAGE OF FIVE TESTS), SEC.
3.15	111
5.4	115
7.2	108

Any test for inflammability is essentially a test to classify materials rather than to determine fundamental properties, and the results of the tests are considered to be reasonably consistent. The two grades of vulcanized fiber are not particularly flame resistant and the data regarding them are included simply to indicate that the method will classify them in comparison with other materials.

The proposed new methods of test for density, ash, zinc chloride, silica and flammability, as appended hereto,[1] are recommended for inclusion in Methods D 229 as Sections 45 to 56, inclusive.

Revision of Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (A.S.T.M. Designation: D 295 - 36 T):[2]

Since power factor has become a criterion for judging insulating materials, a demand has arisen for a test applicable to varnished cloths and varnished cloth tapes. This is provided for in the proposed method of test for power factor and dielectric constant, appended hereto,[3] which are recommended for inclusion in Methods D 295 as Sections 25 to 32, the section numbering for the remainder of the methods to be changed in accordance with the addition of these new sections.

Revision of Tentative Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (A.S.T.M. Designation: D 350 - 35 T):[4]

A number of improvements and revisions in these methods have been prepared. Experience has shown that the range of diameter, as now specified, is insufficient for the larger sizes of varnished tubing. Increases in maximum inside diameters are recommended for sizes Nos. 1 to 15, inclusive. A smoldering test has been developed since varnished tubing is subject to possible injury from the use of soldering irons. Other minor changes involving amplification of the statement on conditioning, greater detail of determination of dimensions, slight alterations in the dielectric strength and heat endurance methods are recommended. These proposed revisions have been incorporated in the completely revised methods appended hereto.[5]

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[1] These changes have been incorporated in the revised tentative methods, see pp. 1038 to 1040.—ED.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1031 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 1016.

[3] These changes have been incorporated in the revised tentative methods, see p. 1047.—ED.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1080 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 1024.

[5] See p. 1056.

Revision of Tentative Methods of Testing Shellac Used for Electrical Insulation (A.S.T.M. Designation: D 411 - 35 T):[1]

Active work was started in 1933 to select or develop a suitable test for flow of shellac. The program of work consisted of three parts, as follows:

1. Selection of suitable and adequate apparatus,
2. Development of proper testing procedure, and
3. Consideration of sample conditioning.

At the time this work was started, there were the following three different types of apparatus available or in use:

1. Inclined metal block,
2. V-shaped glass tube, $\frac{5}{16}$-in. bore, and
3. Straight glass tube, 1-in. bore.

In all of the interlaboratory tests conducted, no less than four grades of shellac having extreme ranges in flow were used and no less than five laboratories participated. In the earlier trials it was evident that the inclined metal block yielded the least consistent results. In view of the fact that it would be an expensive proposition to cut an accurate flow slot in the metal block and to determine by experimentation the proper size and location of the heating elements, it was agreed that glass tubes held much greater possibilities.

Although the V-shaped glass tubes require a very simple structure for support, they were found to be less satisfactory than straight glass tubes of larger bore because of (1) difficulty in cleaning and (2) non-uniformity of heat transfer to specimen.

The large, straight, glass tubes were already in use in the Victor apparatus but the supports and accessories for the tubes were rather expensive and were subject to variation. Two new designs were therefore constructed. Both of these were inexpensive. One of these had greater simplicity but yielded equally reliable results. For this reason, it is illustrated in the appendix to the test methods appended hereto.[2]

Various procedures were tried for determining rate of flow and total flow. The procedure as specified was found to be satisfactory and is in close agreement with those having been used in the industry.

The moisture content of shellac has a great effect on flow. After a 2-yr. study of methods of conditioning samples, it was found that the simple practice of exposing the specimen to the laboratory atmosphere for 24-hr. immediately before testing was more satisfactory than conditioning the specimens or samples in desiccators. It appears that the conditioning of the samples in an air-conditioned room would be the most satisfactory.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1077 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 996.

[2] See p. 1046.

However, since so few laboratories have equipment of this sort, it was considered more practicable to specify the simple expedient stated in the test methods.

The flow property of shellac has been recognized for years as being important in many uses of shellac. The difficulty has been to obtain reliable results with the test methods available. It therefore appears that the proposed methods, appended hereto,[1] should be of help to those who are interested in manufacturing products in which the flow of shellac is involved, such as molded products and certain laminated products.

Revision of Tentative Specifications for Flexible Varnished Tubing Used in Electrical Insulation (A.S.T.M. Designation: D 372 – 35 T):[2]

In order to bring the requirements for the test rod and the table of inside diameters into agreement with the revisions being submitted in the Tentative Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 – 35 T), as given earlier in this report, the following revisions are recommended:

Section 4.—Change the table of inside diameters, appearing in this section, from its present form to read the same as the table given in Section 4 of the revised Tentative Methods D 350.[3]

Section 6.—Change this section to read as follows by the addition of the italicized words and the omission of those in brackets:

6. The number of hours of baking in the heat endurance test at which first cracking of the tubing occurs when bent through 180 deg. over [a rod 0.32 cm. ($\frac{1}{8}$ in.) in diameter] *the rod specified in Section 13 of the Tentative Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (A.S.T.M. Designation: D 350 – 37 T) of the American Society for Testing Materials,*[4] shall be not less than the following:

Grade A	96 hr.
Grade B	72 hr.
Grade C	48 hr.

Revision of Tentative Specifications for Black Bias-Cut Varnished Cloth Tape Used for Electrical Insulation (A.S.T.M. Designation: D 373 – 36 T):[5]

Experience has shown that sewed-seam tapes will not meet the requirements of these specifications. It is accordingly recommended that the following changes, providing for the deletion of all references to such tapes, be made in these specifications:

Section 1.—Change this section to read as follows by the omission of the words in brackets:

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[1] These changes have been incorporated in the revised tentative methods, see p. 1041.—ED.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1003 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 868.

[3] See p. 1057.

[4] See p. 1059.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 912 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 862.

1. These specifications cover black seamless [and sewed-seam] bias-cut varnished cloth tapes 7 to 12 mils in thickness to be used for insulating bus bars, joints and terminals of electric wires and cables, and other electrical apparatus connections.

Note.—Change the Note under Section 1 by the omission of the second item, which reads as follows:

Whether seamless or sewed-seam tapes are desired (Sections 4 and 5).

Section 4.—Change this section to read as follows by the omission of the words in brackets:

4. Splices are those joints made by the manufacturer of the untreated cloth. [In the case of seamless bias-cut tapes] Splices shall not occur more than once in each roll, except that rolls having a length of 6 yd. or less shall contain no splices.

Section 5.—Omit this section, which reads as follows, renumbering the subsequent sections accordingly:

5. Seams are those joints made during or subsequent to the varnish treatment of the cloth. In the case of sewed-seam bias-cut tapes, seams shall not occur more than once in each 45 in. of length.

Section 12.—Change Paragraph (*b*) of this section to read as follows by the omission of the words in brackets:

(*b*) The tensile breaking strength of the "jointed selvages" [in the case of seamless bias-cut tapes,] shall not be less than 80 per cent of the values specified in Paragraph (*a*).

Omit Paragraph (*c*) of this section, which reads as follows:

(*c*) The tensile breaking strength of the "seams," in the case of sewed-seam bias-cut tapes, shall not be less than 60 per cent of the values specified in Paragraph (*a*).

Adoption of Tentative Revision of Standard as Standard

Adoption of Tentative Revision in Standard Methods of Testing Molded Materials Used for Electrical Insulation (A.S.T.M. Designation: D 48 – 33):[1]

The pending tentative revisions[2] of these standard methods are recommended for adoption as standard. Since certain of these revisions have been published for less than a year the committee asks for the necessary nine-tenths vote at the annual meeting in order that they may be referred to letter ballot of the Society for immediate adoption.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1192.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1204 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 1342.

APPENDIX II

SIGNIFICANCE OF TESTS OF ELECTRICAL INSULATING MATERIALS

Significance of the Dielectric Strength Test

By Dean Harvey[1]

Scope of Methods:

The Tentative Methods of Testing Sheet, Tape, and Molded Insulating Materials for Dielectric Strength (D 149 – 36 T)[2] cover the determination of the dielectric strength of these classes of materials. In general, a somewhat similar procedure is followed for the testing of other materials, which tests appear in the following:

D 69 – 36 T.	Tentative Specifications and Tests for Friction Tape for General Use for Electrical Purposes[2]
D 115 – 36 T.	Tentative Methods of Testing Varnishes Used for Electrical Insulation[2]
D 116 – 34.	Standard Methods of Testing Electrical Porcelain.[3]
D 117 – 36.	Standard Methods of Testing Electrical Insulating Oils[3]
D 119 – 35 T.	Tentative Specifications and Tests for Rubber Insulating Tape[4]
D 176 – 37 T.	Tentative Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation[5]
D 348 – 35 T.	Tentative Methods of Testing Laminated Tubes Used in Electrical Insulation[4]
D 349 – 35 T.	Tentative Methods of Testing Laminated Round Rods Used in Electrical Insulation[4]
D 350 – 37 T.	Tentative Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation[6]

Synopsis:

The dielectric strength of an insulating material may be defined as the voltage gradient at which electrical failure or breakdown occurs. It is obtained, for practical purposes, by dividing the breakdown voltage by the thickness of the dielectric between the test electrodes, regardless of whether this value corresponds to the actual maximum voltage gradient that may exist such as at the edges of electrodes or at points of maximum gradient in a non-uniform field. It is thus analogous to the term *tensile strength* as used in mechanical testing.

The test values for dielectric strength of an electrical insulating material vary, to an extent not generally appreciated, with the thickness of the material, the form and size of the electrodes, the time of application of the voltage, the frequency and wave shape of the voltage, the temperature, and the surrounding medium.

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[1] Materials Engineer, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 918, 1059, 1039 (1936); also 1937 Book of A.S.T.M. Tentative Standards, pp. 1002, 1195, 1167.

[3] 1936 Book of A.S.T.M. Standards, Part II, pp. 1178, 1167.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, pp. 1110, 1030, 1026 (1935); also 1937 Book of A.S.T.M. Tentative Standards, pp. 1201, 1033, 1029.

[5] See p. 988.

[6] See p. 1056.

Hence, it usually is not practicable to determine a value for the dielectric strength by means of a laboratory test which will apply accurately under service conditions. Definite numerical values for dielectric strength are needed, however, for specification purposes in the purchase of materials, for comparison of different materials, and for use in design.

When purchasing a material, it is not possible to specify the dielectric strength for the various conditions under which it is to be used, and, therefore, a standard set of procedures is specified to insure the desired quality. The same procedure is followed in comparing different materials.

In making application of materials in design, it must be remembered that the values for dielectric strength determined by the standard procedure will not correspond to those obtained in service unless the conditions of test are the same. As this is not often the case, the test values for dielectric strength usually give only some indication of insulation quality under service conditions.

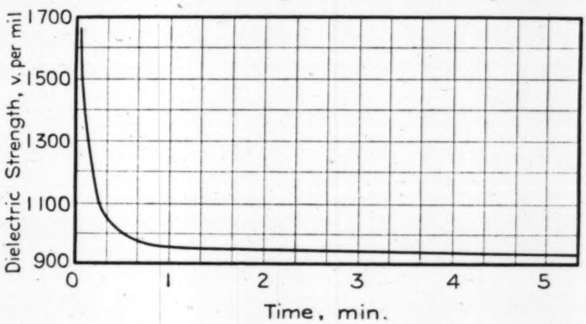

FIG. 1.—Effect of Rate of Increase of Voltage on Dielectric Strength of Black Varnished Cambric.

(Tests made with A.S.T.M. 2-in. flat electrodes with edges rounded to $\frac{1}{4}$-in. radius.)

A brief review of some of the principal factors affecting the values of dielectric strength obtained in test will indicate causes of variation.

Effect of Conditions on Dielectric Strength Tests:

Effect of Time.—The failure of insulation under electrical stress may be due to one or more of the following causes:

1. Generation of heat within insulation, due to absorption of energy,
2. Chemical action, producing oxygen, etc., by ionization of the air or lowering the resistivity with increase in energy loss, and
3. Mechanical disruption, due to stresses set up by the electrostatic field, or to gases suddenly produced within the material.

The first two of these are progressive and involve the element of time.

Rate of Application of Voltage.—The dielectric strength of insulating materials varies greatly with the rate of application of the voltage, that is, the more rapid the increase in voltage, the higher will be the breakdown voltage. This is illustrated by typical curves shown in Fig. 1. Three standard methods for the application of voltage are in general use:

A. The Short-Time Dielectric Strength Test in which the voltage is increased uniformly to breakdown at a specified rate.

B. The One-Minute Step-by-Step Dielectric Strength Test in which the voltage is held for 1 min. at a selected starting voltage followed by 1-min. steps at higher voltages, increasing successively by regular increments, and

C. The Endurance or Long-Time Dielectric Strength Test in which the voltage is held either until failure or for a definitely long period of time, followed by equal periods of time at higher voltages, increasing successively by regular increments. A 30-min. endurance time is the standard procedure in materials testing on taped samples of varnished cloth, and 5 min., 15 min. and longer time periods up to 8 hr. or more in cable practice.

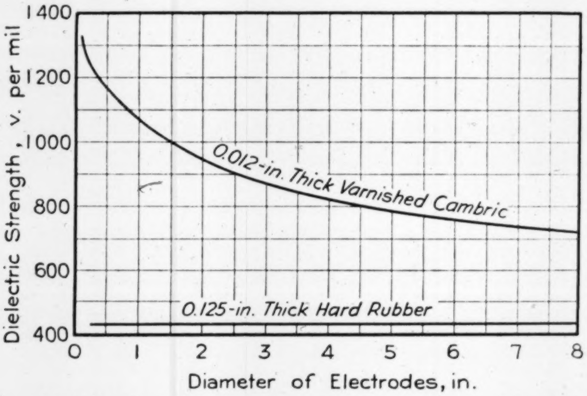

FIG. 2.—Relation of Dielectric Strength to Area of Electrodes for Varnished Cambric and Hard Rubber.

The short-time test does not give as good an indication as does the step-by-step test of what the dielectric strength will be when the voltage is applied for a long period of time.

Effect of Electrodes.—The dielectric strength decreases with increase in the diameter of the electrode, this condition being most pronounced with thin sheets and negligible with thick sheets, as illustrated by typical curves in Fig. 2. Electrodes 2 in. in diameter with corners rounded to a radius of $\frac{1}{4}$ in., thus having an effective diameter of $1\frac{1}{2}$ in. were selected as a compromise. They provide tests near enough to the flat portion of the curve to avoid large variations in tests and yet small enough not to require an excessive amount of material for test. For narrow specimens, electrodes $\frac{1}{4}$ in. in diameter with corners having $\frac{1}{32}$-in. radius are used since they give higher test values than the 2-in. electrodes.

Thickness of Specimen.—In general, the dielectric strength per unit thickness of specimen decreases with increase in thickness, being proportional to the two-thirds to three-quarters power of the thickness.

Frequency.—While the dielectric strength decreases with increase in frequency, the increase over the range up to 100 cycles is very small and may be considered negligible. When materials are to be used at audio or radio frequencies, the dielectric strength should be determined at the frequency to be used. The dielectric strength at radio frequencies may be only a very small fraction, ranging from 10 to 20 per cent of that at 60 cycles.

Temperature.—In general, the dielectric strength decreases with increase in temperature, but the extent to which this occurs varies greatly with different materials. When materials are to be used at other than room temperature, their dielectric strength should be determined over their operating range of temperature. If this range is considerable, tests should be made at not less than four different temperatures, including the maximum and the minimum and a curve plotted of volts per mil *versus* temperature.

Conditioning.—The dielectric strength of electrical insulating materials is considerably affected by the presence of moisture in the materials. It is, therefore, necessary to condition materials that adsorb moisture. Methods of conditioning are being studied by Committee D-9.

SIGNIFICANCE OF THE RESISTIVITY TEST

BY H. L. CURTIS[1]

Synopsis:

Resistivity is that property of a material which determines its suitability for use as an insulator. If the suitability of an insulating material for a particular application is to be investigated, the surface resistivity as well as the volume resistivity may be required. If the material absorbs moisture, its suitability as an insulator cannot usually be determined by resistivity measurements. For such absorbent materials measurements are made of the insulation resistance on a specimen in a form similar to that which is to be used in the application so that it can be subjected to the same conditions as will be encountered in service.

Scope of Methods:

The Standard Methods of Test for Resistivity of Insulating Materials (D 257 – 33)[2] cover the determination of surface resistivity and volume resistivity of both solid and liquid insulating materials. A proposed revision of this method, issued in the form of Tentative Methods of Test for Insulation Resistance of Electrical Insulating Materials (D 257 – 37 T),[3] covers the determination of insulation and volume resistance and approximate surface resistance of electrical insulating materials; also means of converting the latter two into terms of volume and surface resistivities.

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[1] Principal Physicist, National Bureau of Standards, Washington, D. C.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 1203.

[3] See p. 1016.

Resistivity of Insulators:

An electrical insulating material is ordinarily defined as a material which has a high resistivity. Hence, a measurement of the resistivity of a material is necessary to determine whether it is an insulator. From this point of view, resistivity is the most fundamental quality of a material to be used as an insulator. If the volume resistivity of a material is less than one megohm-centimeter, it will not normally be considered an insulating material. However, the resistivity of most insulating materials is very much higher than the above minimum. Approximate values for some common materials are given in Table I.

TABLE I.—VOLUME RESISTIVITIES OF SOME COMMON SOLID INSULATORS.

MATERIAL	VOLUME RESISTIVITY, MEGOHM-CENTIMETERS
Ivory	200
Celluloid	20 000
Marble	100 000
Plate glass	20 000 000
Amber	50 000 000 000
Fused quartz	5 000 000 000 000
Ceresin	more than 5 000 000 000 000

Resistivity Measurement:

The resistivity of a material is defined as the reciprocal of the current between two electrodes placed on opposite faces of a centimeter cube of the material when there is unit potential difference between the electrodes, provided the current distribution is uniform throughout the cube, and the time of application of the voltage has been sufficiently long so that the current is constant. It is sometimes desirable to designate the resistivity, defined in this manner, as the volume resistivity in order to distinguish it from cases in which the current distribution is not uniform. While defined in terms of a centimeter cube, actual measurements are generally made on specimens of such size and shape that a guard ring can be employed to insure that the measured current is in the body of the material.

The extreme case of lack of uniformity of current distribution occurs in some solids in which the current between the electrodes, on the opposite faces of a cube of the solid, is practically confined to a thin layer on the surface. This surface layer is generally water which has been adsorbed from the moisture of the surrounding air, and which may have a conductivity many millions of times that of the solid insulation. For those materials in which the current through the surface film can be considered independently of the current through the body of the material, the resistance between two opposite edges of a square of the surface film is called the surface resistivity.

In the more general case, the current distribution over a cross-section of a cube of finite size having electrodes on opposite faces is an unknown function of the distance from the surface of the cube. In such a case there is no possibility of using measurements of resistance between one set of electrodes attached to a sample to predict the value of the resistance between electrodes of a decidedly different form and size which are attached to the same sample. Since the resistivity cannot be determined for materials in which the current distribution conforms to this general case, the insulation resistance of a specimen of some arbitrarily selected size and shape is measured. This may serve to differentiate between materials but does not give a measure of resistivity.

Surface Resistivity:

The surface resistivity of a material is determined by the films of water, oil or other liquid on the surface; of these the water films are of greatest importance. For most applications, the surface will be exposed to the air, and hence, for all materials except the waxy ones, moisture from the air will be adsorbed on the surface, the amount depending on the relative humidity. The atmospheric moisture dissolves some of the salts which are always present on any surface that has not been recently cleaned, thus making a conducting layer on the surface of the insulator. The thickness of this film depends on the humidity of the surrounding air, and consequently the surface resistivity varies with the humidity. This is illustrated by the values given in Table II for the same materials for which the volume resistivity has been given. At 25 per cent relative humidity there is very little surface film so that the surface resistivity is high and its measurement is difficult with materials having a low volume resistivity. At 90 per cent relative humidity the surface films are relatively thick, so that the surface resistivities are readily determined. It will be seen that a material which has a high volume resistivity does not necessarily have a high surface resistivity.

TABLE II.—SURFACE RESISTIVITIES OF SOME COMMON SOLID INSULATORS.

	SURFACE RESISTIVITY, MEGOHMS	
MATERIAL	AT 25 PER CENT RELATIVE HUMIDITY	AT 90 PER CENT RELATIVE HUMIDITY
Ivory	10 000	50
Celluloid	100 000	1 000
Marble	100 000	10
Plate glass	100 000 000	20
Amber	1 000 000 000	100 000
Fused quartz	1 000 000 000	100
Ceresin	100 000 000 000	100 000 000 000

Volume Resistivity of Liquids:

The volume resistivity of any insulating liquid which has been carefully purified is very high. However, most commercial liquids have sufficient dissolved water to lower the resistivity appreciably. A value of 100,000,000 megohm-centimeters is about as high as is found in transformer oils. There is some indication that the volume resistivity is directly related to the amount of dissolved water, but the equation for expressing this relationship has not been established.

Purpose of Resistivity Measurements:

Many of the measurements of volume resistivity are made for the purpose of judging the uniformity of a material or of estimating the amount of impurities in a material. Hence, many specifications state that the resistivity shall lie within certain limits or shall not be less than a certain amount, although the actual value of the resistivity is of no importance in the application for which the material is intended. Such requirements are generally based on the experience of the consumer with a particular material, but this experience may not be applicable to a different type of material.

Many of the applications that require values of resistivity for use in design formulas call for solid insulators where the leakage current is determined by both the volume and surface resistivity. Moreover, it is generally essential that the leakage current shall not exceed a certain value even under the most unfavorable weather conditions, which are generally considered to involve a relative humidity of 90 per cent. For such an application, surface resistivity is generally more important than volume resistivity.

Conditioning:

Conditioning may be necessary for measuring the resistivity of materials that adsorb moisture. Specimens on which surface resistivity is to be measured are generally kept for two or three days at a definite temperature and humidity before the measurements are started. The surface resistivity of a material has no meaning unless the humidity of the surrounding air is stated, and even this may not be sufficient, for surface films do not respond quickly to changes in humidity and temperature. Conditioning is also required in determining the volume resistivity of materials that absorb moisture. Since water penetrates some materials very slowly, the volume resistivity of absorbing materials is not usually measured, but the insulation resistance determined for a specimen which has been immersed in water for some arbitrarily selected period, such as a day, week, or month.

Precision in Measurement:

The accuracy required in measurements of resistivity is very small, since the values for two samples of the same material generally differ by at least 50 per cent and frequently by several hundred per cent. Hence, the mechanical dimensions required in calculating the resistivity are easily measured, as is also the resistance except when it is very high. Because of the ease of measurements, results are frequently given with an accuracy of 1 per cent, but there are few cases where more than one significant figure has any meaning. The greatest difficulties in determining the resistivities of a material are the selection of representative samples of the material, and in so attaching electrodes to these samples that the volume and surface resistivities can be independently evaluated. In measuring surface resistivity, the humidity of the surrounding air must be carefully controlled, since in some cases a change in relative humidity of 5 per cent may change the surface resistivity by a factor of ten. At least two samples should be measured for every material to give an idea of its uniformity.

SIGNIFICANCE OF THE IMPACT TEST

BY M. P. DAVIS[1]

Scope of Methods:

The Tentative Methods of Testing Electrical Insulating Materials for Resistance to Impact (D 256 – 34 T)[2] are intended to cover the determination of the relative toughness of electrical insulating materials as indicated by the energy used in breaking a standard test specimen in one blow.

The purpose of the notched bar impact test is to show the resistance of the material to shock loads. The basic assumption of the test is that resistance to shock depends upon the ability of materials rapidly to equalize dangerously concentrated stresses by flow or deformation without failure. The notch and the speed of test combine to produce a concentration of stress as a test condition.

There is no known correlation between the various static tests such as compression, tension, flexure, etc., and the impact test. Several materials may show the same values on static tests (compression, tension, flexure, etc.), and from the results, it may be difficult to differentiate between them, while the impact test may give decidedly different values.

For a more complete discussion of this test, reference should also be made to the Tentative Methods of Impact Testing of Metallic Materials (A.S.T.M. Designation: E 23 – 34 T)[3] and the Appendix to these Tentative Methods which contains information on the applications of the impact test and its relation to service conditions. Information here given applies equally to molded materials as it does to metallic materials, with the following exceptions:

(*1*) The recommended test specimen for molded materials is $\frac{1}{2}$ by $\frac{1}{2}$ in. square instead of 10 by 10 mm. square, and

(*2*) The notch is the same as the notch used for the metallic test specimen, except that for molded materials the notch is 0.100 in. deep.

Tentative Methods E 23 – 34 T, under Procedure, Section 9 (*a*) states that a machine of 120-ft-lb. capacity is used for the testing of metallic materials. Molded insulation does not require a machine of this capacity and it should be tested on a machine of a capacity of approximately 2 ft-lb.

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[1] Chief Chemist, Otis Elevator Co., Yonkers, N. Y.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 995 (1934); also 1937 Book of A.S.T.M. Tentative Standards, p. 1023.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 1201 (1934); also 1936 Book of A.S.T.M. Tentative Standards, p. 1280.

REPORT OF COMMITTEE D-11
ON
RUBBER PRODUCTS

Committee D-11 on Rubber Products has held one meeting during the year in addition to the one held at the 1936 annual meeting. This was at Chicago, Ill., on March 4, 1937, in connection with the spring group meetings of A.S.T.M. committees.

During the past year the committee has lost two members by death, Mr. C. W. Mowry and Mr. J. E. Chapin, both of whom have been closely associated with the work of the committee for many years. Their presence at meetings and willing cooperation in the work of the committee will be greatly missed. Mr. N. J. Thompson has been designated to succeed Mr. Mowry, representing the Associated Factory Mutual Fire Insurance Cos. In addition, the committee has lost by resignation, Messrs. Frank Zeleny, C. B. & Q. R. R.; E. H. Grafton; H. W. Asire, General Motors Corp.; and W. R. Marsden, Kelly-Springfield Tire Co. Also, Messrs. Stanley Krall and J. E. Partenheimer of the Fisk Rubber Corp. have been replaced by Mr. W. J. Geldard. Mr. L. J. DeHolczer, of Jenkins Bros., has been replaced by Mr. G. P. Mooshegranz.

New members who have been added to the committee during the year include the following: Messrs Alfred Bellis, John A. Roebling's Sons Co.; D. E. Baker, Montsanto Chemical Co.; H. S. Karch, Lima Cord Sole and Heel Co.; W. H. Reynolds, American Instrument Co.; B. J. Barmack, Commonwealth Edison Co.; and A. W. Staller, Crescent Insulated Wire and Cable Co.

Additional company representatives have been added as follows: Messrs. J. E. McGraw, General Electric Co.; David C. Scott, Jr., Henry L. Scott Co.; M. G. Shepard, U. S. Rubber Co.; R. V. Yohe, The B. F. Goodrich Co. The representation of the National Electrical Manufacturers Association in the committee has also been altered by the designation of Mr. E. D. Youmans as principal in place of Mr. C. R. Boggs, and by the addition of Mr. E. W. Schwartz.

At the annual meeting last year organization was authorized of a new Subcommittee XXI on Tests of Liquid Rubber Products, including such materials as rubber cements and products made from latex or involving water dispersions of rubber. Mr. L. M. Freeman has been appointed chairman of this subcommittee.

In compliance with the new by-laws of the committee, which were adopted last year, the Advisory Subcommittee has been reorganized to consist of the active officers and four others elected by the committee. The members so elected are Messrs. R. A. Schatzel, H. M. Frecker, M. E. McDonnell and P. L. Wormeley.

Subsequent to the 1936 annual meeting the committee presented to the Society, through Committee E-10 on Standards, revisions of the Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 – 35 T) and for Insulated Wire and Cable: Performance Rubber Compound (D 353 – 35 T).

These revisions included principally changes in permissible variations in size of conductors, shielding, thickness of insulation and extension of the table of the alternating test voltages to include rated circuit voltages up to 8000 v., provision for flame-proof finish, and bending diameters in the bend test and the moisture absorption test. These revisions were thoroughly considered and unanimously approved by the committee and are intended to increase the usefulness of the specifications and bring them in line with modern practice. The revisions were accepted[1] by Committee E-10 on August 26, 1936, and the specifications in their revised form appear in the 1936 *Proceedings*.[2]

RECOMMENDATIONS AFFECTING STANDARDS

I. *Proposed Tentative Standards:*

Proposed Tentative Method for Air Pressure Heat Test of Vulcanized Rubber.—This method is the result of the work of Subcommittee XV on Life Tests for Rubber Products, and was discussed in the 1936 report[3] of the Committee. Although the procedure which involves subjecting rubber specimens to the action of compressed air at elevated temperature is similar in many respects to the standard aging tests for rubber, this method can hardly be considered to be an aging test suitable for evaluating the life of all types of rubber compounds under normal conditions because the temperature employed, 260 F., is so high as to be in the usual vulcanizing range. The method is, therefore, designated as a heat test rather than an aging test and is considered to be very useful for service evaluation of rubber compounds designed to resist heat and oxidation. The short time required for securing marked deterioration of vulcanized rubber under the conditions of the test adds greatly to the convenience and utility of the method when it is necessary to obtain quickly an estimate of the relative stability of vulcanized rubber under severe conditions. Tests of this type have come into quite general use but heretofore have undergone very little standardization. The committee accordingly recommends that the proposed Tentative Method for Air Pressure Heat Test of Vulcanized Rubber, as appended hereto,[1] be accepted for publication as tentative.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-11 reported results of the letter ballot vote as follows: Of a total membership of 83, 36 members returned their ballots, of whom 28 voted affirmatively, 0 negatively, and 8 members marked their ballots "not voting."

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 1065, 1092 (1936); also 1936 Book of A.S.T.M. Tentative Standards, pp. 1059, 1086.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 488 (1936).

II. Proposed Revisions of Tentative Standards:

The committee is proposing revisions of two tentative methods of test and two tentative specifications, as indicated below, and recommends that these tentative standards as revised be continued as tentative:

Tentative Methods of Test for Abrasion Resistance of Rubber Compounds (D 394 – 34 T).[2]—The following revision in "Method B, E. I. du Pont de Nemours and Co. Abrader," is recommended so that the method will be in agreement with generally accepted practice:

Section 10.—Change the third sentence to read as follows by the addition of the italicized words and figure and the omission of those in brackets:

> Two test specimens are mounted on the inside of a bar, one placed at each end [and at] *so that their centers are* a distance of [4.5] *5* in. apart.

Tentative Methods of Test for Compression Set of Vulcanized Rubber (D 395 – 36 T).[3]—Objections were received by Subcommittee XVII with respect to the permissible tolerance in the thickness dimension of the standard test specimen, indicating that it might be closer than is necessary and difficult of attainment in some instances. Accordingly, an investigation was made in the subcommittee involving an extensive series of tests, using duplicate samples of varying thicknesses. Comparison of the results from samples having thickness equivalent to the plus tolerance with those from samples at the minus tolerance showed that the differences encountered are no greater than often occur when using individual samples of the same compound and the same thickness. The committee considers that it has proven the former tolerance to be unnecessarily close and accordingly recommends that this section be revised as follows:

Section 9.– Change the fourth sentence to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> When necessary, the specimen thickness shall be reduced to the required ½ in. by cutting transversely with a sharp knife and carefully buffing to size within a permissible variation of [±0.005 in.] *+0.005 in. and –0.025 in.*

Tentative Specifications for Insulated Wire and Cable: Class AO, 30 Per Cent Hevea Rubber Compound (D 27 – 36 T).[4]—The revisions[5] that are being proposed in these specifications include: (1) an extension of Table V on

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[1] See p. 1122.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 1150 (1934); also 1936 Book of A.S.T.M. Tentative Standards, p. 1144.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1133 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 1137.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1065 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 1059.

[5] Subsequent to the annual meeting additional revisions of these specifications were presented to Committee E-10 on Standards, see Editorial Note, p. 453. These have been incorporated in the revised tentative specifications, see p. 1070.—ED.

Insulation Resistance to cover the same range as Table IV on Test Voltage; (2) limitation of the size of cross-section of test specimens in tensile strength test in order to provide greater accuracy; (3) editorial changes covering details of the electrical testing of multiple conductor cable to make these more specific; and (4) elimination of the moisture absorption test for braids as it has proven to be unsatisfactory.

Table V.—Add insulation resistance values in megohms for the conductor sizes shown with rubber insulation thicknesses from $\frac{13}{64}$ in. to $\frac{17}{64}$ in., incl., as shown in the accompanying Table I.

TABLE I.—INSULATION RESISTANCE, MEGOHMS—1000 FT. AT 60 F. (15.5 C.)

Size of Conductor, A. w. g. Numbers or Circular Mils	Insulation Thickness in Sixty-fourths of an Inch				
	13	14	15	16	17
No. 8	13 100	13 600	14 100	14 600	14 900
No. 6	10 650	11 150	11 500	12 000	12 450
No. 4	9 300	9 700	10 100	10 500	10 900
No. 2	7 950	8 400	8 800	9 150	9 500
No. 1	7 350	7 650	8 000	8 400	8 750
No. 0	6 750	7 050	7 400	7 800	8 100
No. 00	6 200	6 500	6 900	7 150	7 500
No. 000	5 700	6 150	6 350	6 650	6 900
No. 0000	5 200	5 500	5 800	6 050	6 350
250 000 circular mils	4 900	5 200	5 450	5 750	5 950
350 000 circular mils	4 250	4 500	4 700	4 950	5 200
500 000 circular mils	3 700	3 950	4 150	4 350	4 550
750 000 circular mils	3 100	3 300	3 500	3 700	3 900
1 000 000 circular mils	2 750	2 950	3 100	3 300	3 450
1 250 000 circular mils	2 500	2 650	2 850	3 000	3 150
1 500 000 circular mils	2 300	2 450	2 600	2 700	2 950
1 750 000 circular mils	2 150	2 300	2 450	2 600	2 750
2 000 000 circular mils	2 050	2 200	2 300	2 450	2 600

Section 26.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

26. (a) Size.—The test specimen may be the entire section of the insulation in the case of small wires, [or] *but* in the case of [a large] wire [or] *and* cable [either a segment of a section cut with a sharp knife held tangentially to the conductor or a shaped specimen cut out with a standard die] *of No. 6 A.w.g. and larger or with an insulation thickness greater than* $\frac{3}{32}$ *in., samples approximately square in section shall be cut from the insulation with a cross-section not greater than 0.025 sq. in.* The test specimen shall be as free as possible from surface incisions and imperfections. [There shall be no limit to the cross-section of the test specimen, except as restricted by the capacity of the testing machine.]

Change Paragraph (*c*) of this section to read as follows by the addition of the italicized words and the omission of the words and figure in brackets:

(c) Buffing.—In the event of any irregularities on the surface of the test specimen *including corrugations from large strands,* it shall be made smooth and of uniform thickness [within 5 per cent of the original thickness] by buffing [except when large strands are used, in which case the rubber sample shall be buffed sufficiently to remove all corrugations].

Section 49.—To the end of Paragraph (*b*) add a new paragraph to read as follows:

> Single-conductor cables shall be tested between conductor and sheath or water in which they are immersed at the test voltage required by Table IV.

To the end of Paragraph (*c*) of this section add a new paragraph to read as follows:

> Multiple conductor cables after assembly shall be tested at the test voltage required by Table IV.

Section 50.—Omit this section which describes the application of voltage on single-conductor and multiple-conductor cables.

Table IX.—Change the minimum thickness requirements for braid in Table IX by the omission of the fourth decimal place. The revised requirements will read as follows:

MINIMUM THICKNESS OF BRAID, IN.
0.016
0.017
0.020
0.026
0.032

Table X.—Change the minimum thickness requirements for braid in Table X by the omission of the fourth decimal place. The revised requirements will read as follows:

MINIMUM THICKNESS OF BRAID, IN.
0.020
0.022
0.026
0.032
0.037
0.045
0.056

Section 66.—Omit this section which describes the moisture absorption test.

Tentative Specifications for Insulated Wire and Cable: Performance Rubber Compound (D 353 – 36 T).[1]—The following revisions[2] proposed in these specifications correspond to the changes recommended above in Specifications D 27 – 36 T, and are intended to keep the two specifications consistent with each other:

Table II.—Make the same changes in this table as proposed above in Table V of Specifications D 27 – 36 T.

Section 10.—Replace the present Paragraphs (*a*) and (*c*) of this section by new paragraphs as proposed above for Paragraphs (*a*) and (*c*) of Section 26 of Specifications D 27 – 36 T.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1092 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 1086.

[2] Subsequent to the annual meeting additional revisions of these specifications were presented to Committee E-10 on Standards, see Editorial Note, p. 453. These have been incorporated in the revised tentative specifications, see p. 1109.—ED.

Section 32.—Add new paragraphs at the end of Paragraphs (*b*) and (*c*) of this section as proposed above for Paragraphs (*b*) and (*c*) of Section 49 of Specifications D 27 – 36 T, referring to the table as Table I.

Section 33.—Omit this section which describes the application of voltage on single-conductor and multiple-conductor cables.

III. Withdrawal of Tentative Standard:

Tentative Specifications for Insulated Wire and Cable: Class A, 30 Per Cent Hevea Rubber Compound (D 393 – 34 T).[1]—Withdrawal of these specifications has been contemplated for some time as the type of rubber compound covered has become obsolete and is now used very little. These specifications which were issued in 1934 have been almost entirely supplanted by others now published as tentative. The committee accordingly recommends that the specifications be discontinued.

IV. Tentative Standard Continued as Tentative:

Tentative Specifications for Rubber Pump Valves (D 151 – 31 T).—These specifications, which have already been continued as tentative for a number of years, are not yet suitable for adoption as standard. These specifications are in general agreement with the Federal Specifications for similar products but the committee is now inclined to feel that revisions are in order in both of these and is unwilling to recommend their adoption pending such changes. It is therefore desired that these specifications be continued as tentative for the present.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 84 members, the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. PROPOSED TENTATIVE STANDARD			
Method for Air Pressure Heat Test of Vulcanized Rubber	50	2	11
II. REVISIONS OF TENTATIVE STANDARDS			
Methods of Test for Abrasion Resistance of Rubber Compounds (D 394 – 34 T)	54	0	9
Methods of Test for Compression Set of Vulcanized Rubber (D 395 – 36 T)	54	0	9
Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 – 36 T)	47	0	16
Specifications for Insulated Wire and Cable: Performance Rubber Compound (D 353 – 36 T)	47	0	16
III. WITHDRAWAL OF TENTATIVE STANDARD			
Specifications for Insulated Wire and Cable: Class A, 30 per cent Hevea Rubber Compound (D 393 – 34 T)	39	0	20

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 1126 (1934); also 1936 Book of A.S.T.M. Tentative Standards, p. 1099.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Mechanical Rubber Hose (E. G. Kimmich, chairman). —This subcommittee has decided to undertake a consolidation and amplification of the two existing A.S.T.M. Standards, D 379 and D 380, on testing rubber hose. Although these methods in their present form have been fairly satisfactory, it has been found they are not sufficiently comprehensive to cover the field thoroughly because of the wide variety of types and constructions of hose which are in common use. It is believed that their utility can be effectively increased by combining them into a single set of test procedures and by supplementing the present methods with additional tests required for types of hose not covered at present. The new proposed standard[1] will be recommended to replace the two present methods.

The subcommittee also plans to submit a tentative revision of the Standard Specifications for Cotton Rubber-Lined Fire Hose for Public and Private Fire Department Use (D 296 – 35), recommending the omission of detailed descriptions of methods of test and substituting suitable reference to the new general methods of test for rubber hose and to the Methods of Chemical Analysis of Rubber Products (D 297 – 36). In this way, extensive duplication in the standards will be avoided and some inconsistencies will be eliminated, particularly those existing at present in the hydrostatic test procedures.

Subcommittee IV on Protection of Persons from Electrical Shock (H. S. Vassar, chairman).—This subcommittee expects to recommend, in the near future, revisions in the Standard Specifications for Rubber Gloves (D 120 – 23). A survey which has been completed indicates that the present specifications are being employed by approximately 70 per cent of the buyers of rubber gloves. However, after the 14 years during which the present specifications have been standard, some changes are necessary to bring them up to date. The major changes contemplated include the introduction of an accelerated aging test and increase in tensile strength, elongation and set requirements. A number of minor revisions and editorial changes are also being considered. All of these proposals have been submitted to a considerable number of producers and consumers for comment, and, so far, the replies indicate that there is little, if any, demand for any more extensive changes than those proposed. It is anticipated that these revisions may be completed so as to be presented to the Society during the coming year through Committee E-10 on Standards.[2]

Subcommittee V on Insulated Wire and Cable (R. A. Schatzel, chairman). —In addition to the revisions being recommended in the two tentative specifications for insulated wire and cable (D 27 – 36 T and D 353 – 36 T)[3] as mentioned earlier in this report, this subcommittee has been preparing specifications covering tough rubber sheath compounds for cables and for heat-resisting compounds.[1] Tentative drafts of these specifications have been circulated and it is expected that the final recommendations will be available in the near future. In addition, the subcommittee contemplates separation of the test methods for insulated wire and cable from the present specifications and their issuance in the form of a separate standard. It is believed that this will increase the utility of both the specifications and the methods and will avoid some duplication. A tentative draft of the proposed standard test methods is under consideration.[2]

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[1] Subsequent to the annual meeting, Committee D-11 presented these methods to the Society through Committee E-10 on Standards, see Editorial Note, p. 453.—ED.

[2] See Editorial Note, p. 453.—ED.

[3] Subsequent to the annual meeting, Committee D-11 presented additional revisions of these specifications to the Society through Committee E-10 on Standards, see Editorial Note, p. 453.—ED.

Subcommittee X on Physical Testing of Rubber Products (H. M. Frecker, chairman).—This subcommittee plans to extend the work of the abrasion subcommittee on development of standard rubber test specimens having certified properties into the field of tensile measurements. Progress in a program of this sort is necessarily slow but it is expected that ultimately reference standard samples will be made available as a result of the work of the two subcommittees, in consequence of which marked improvement in the duplicability and accuracy of physical tests of rubber will result. Committee D-11 has also authorized the formation of a section of Subcommittee X to act in an editorial capacity on recommended standards and on matters pertaining to the publication of the compilation of A.S.T.M. Standard Methods of Test for Rubber Products. It is hoped that the publication of this manual can soon be put on an annual basis.

Subcommittee XVII On Rubber Products for Absorbing Vibration (J. J. Allen, chairman).—During the extensive investigation already mentioned of the effect of sample thickness in the Tentative Methods of Test for Compression Set of Vulcanized Rubber (D 395 – 36 T), the subcommittee also studied a proposed modification of the test wherein the decay in load instead of change in dimensions during the action of compression and heat aging was taken as the measure of compression set. The purpose of this work was to endeavor to find a more accurate method of evaluation than thickness measurements, which are often difficult to carry out precisely because of warping of the specimen during compression and heat treatment. As yet, the proposed modification seems to present formidable difficulties, though further study and some changes in technique may improve the situation.

Subcommittee XIX on Tests for Properties of Rubber and Rubber-Like Materials in Liquids (O. M. Hayden, chairman).—Following the survey reported in 1935[3] on methods of test for resistance of rubber and rubber-like materials to action of solvents, this subcommittee investigated some of the factors affecting the rate of deterioration of the specimens in such tests.

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[1] Subsequent to the annual meeting, Committee D-11 presented these specifications to the Society through Committee E-10 on Standards, see Editorial Note, p. 453.—ED.

[2] Subsequent to the annual meeting, Committee D-11 presented these methods to the Society through Committee E-10 on Standards, see Editorial Note, p. 453.—ED.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 427 (1935).

In a cooperative test program using uniformly prepared samples of different size and shape tested in five different laboratories, in water, benzene, and kerosine, the subcommittee found (1) the laboratories checked closely using kerosine and fairly well in benzene but poorly with water; (2) increase in volume during immersion in liquids is in inverse ratio to the thickness of the test specimen; (3) the greater the surface exposure the faster the rate of swelling; (4) slight buffing of the surface increases the rate of swelling; (5) volume increase measurements are in close agreement whether determined by the water displacement method or computed from increase in weight and specific gravity determinations; (6) if the volume increase is determined by calculation from the change in one dimension the direction of grain of the specimen has a marked influence on results. After consideration of these and many other factors involved in the test, the subcommittee has agreed on the following details for a standard method: (1) size of test pieces; (2) liquid to be same as in actual service; (3) equipment; (4) methods of measurement; (5) conditions of test. It is expected that the methods will be completed so that they may be presented to the Society for publication as tentative through Committee E-10 on Standards during the summer.[1]

Subcommittee XXI on Tests of Liquid Rubber Products (L. M. Freeman, chairman).—This subcommittee held its organization meeting in March and decided that for the immediate future it would devote its attention to the formulation of standard methods of test for the evaluation of rubber cements and rubber latex. The work on cements will include standardization of tests for viscosity and for the determination of total solids. A cooperative test program has been outlined involving a survey and study of the various test procedures in use at present. A cooperative series of tests on latex by interested laboratories is also planned. The latex will be obtained from one source of supply and will include latices having both normal and 60 per cent rubber concentration. Methods will be investigated for determining color, total solids, dry rubber content, ammonia, and hydrogen ion concentration.

This report has been submitted to letter ballot of the committee which consists of 84 members; 63 members returned their ballots, of whom 57 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

O. M. HAYDEN,
Chairman.

ARTHUR W. CARPENTER,
Secretary.

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[1] See Editorial Note, p. 453.—ED.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-11 on Rubber Products presented to the Society on August 26, 1937, through Committee E-10 on Standards the following recommendations:

Proposed Tentative Specifications for:

Rubber Gloves for Electrical Workers on Apparatus or Circuits not Exceeding 3000 Volts to Ground, to replace immediately Standard Specifications D 120 - 23, see p. 1062.

Insulated Wire and Cable: Heat-Resisting Rubber Compound, see p. 1096.

Proposed Tentative Methods of:

Testing Rubber Hose, to replace immediately Standard Methods D 379 - 36 and D 380 - 36, see p. 1153.

Testing Rubber Insulated Wire and Cable, see p. 1166.

Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids, see p. 1148.

Proposed revision of Tentative Specifications for:

Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 - 37 T), see p. 1070.

Insulated Wire and Cable: Performance Rubber Compound (D 353 - 37 T), see p. 1109.

Withdrawal of Standards:

Specifications for Rubber Gloves for Electrical Workers on Apparatus or Circuits not Exceeding 3000 Volts to Ground (D 120 - 23),

Methods of Test for Rubber Hose—Braided Construction (D 379 - 36), and

Methods of Test for Rubber Hose—Wrapped Construction (D 380 - 36).

The above recommendations were accepted by Committee E-10 and appear in this volume on the pages given above.

REPORT OF COMMITTEE D-12
ON
SOAPS AND DETERGENTS

As a result of extensive studies by a special committee appointed in 1935 by the Executive Committee of the Society to consider the desirability of forming an A.S.T.M. standing committee on soap, a new standing committee on Soaps and Detergents was formally organized at a meeting held on July 1, 1936, in Atlantic City, N. J., during the annual meeting of the Society. The scope of the committee is as follows: Soaps and detergents including the materials entering into their manufacture. A set of committee regulations was adopted.

At the organization meeting, officers of the committee were elected, as follows: Chairman, H. P. Trevithick; Vice-Chairman, F. W. Smither; Secretary, B. S. Van Zile. An Advisory Committee was chosen, consisting of the officers of the committee and the following members: J. B. Crowe, J. E. Doherty, Ephraim Freedman, G. E. Hopkins, Frederick Krassner, and Pauline B. Mack.

The following subcommittees and sections have been appointed:

Subcommittee I on Methods of Testing (J. B. Crowe, chairman)
  Section A on Soaps (M. L. Sheely, chairman)
  Section B on Sulfonated Detergents (Ralph Hart, chairman)
  Section C on Dry Cleaning Soaps (G. G. Gaubatz, Jr., chairman)
  Section D on Special Detergents (J. D. MacMahon, chairman)

Subcommittee II on Specifications (F. W. Smither, chairman)
  Section A on Textile Soaps (G. E. Hopkins, chairman)
  Section B on Built Soaps (Frederick Krassner, chairman)
  Section C on Straight Soaps (Israel Katz, chairman)
  Section D on Dry Cleaning Detergents (G. G. Gaubatz, Jr., chairman)
  Section E on Sulfonated Detergents (J. B. Crowe, chairman)
  Section F on Special Detergents (C. C. Zeigler, chairman)
  Section G on Metal Cleaners (G. B. Hogaboom, chairman)

Subcommittee III on Nomenclature and Definitions (Frederick Kenney, chairman)

G. E. Hopkins resigned as chairman of Section A of Subcommittee II on March 1 and was succeeded by C. L. Nutting.

In addition to the organization meeting, the committee held two other meetings during the year, on December 3, 1936, and on March 29, 1937, both in New York City with an attendance of 46 and 40, respectively. At the December meeting the Standard Methods for the Sampling and Analysis of Soap and Soap Products of the American Oil Chemists' Society were reviewed and approved for submission to the A.S.T.M. for publication as tentative. These methods have also been adopted by the American Chemical Society.

At the spring meeting of the committee reports were received from the subcommittees and sections recommending certain specifications and definitions for submission to the Society for publication as tentative, as given below. There were also submitted Proposed Methods of Chemical Analysis of Sulfonated Oils which are published in Appendix I to this report as information only for the purpose of obtaining criticisms and constructive suggestions for further improvements. Proposed Methods of Chemical Analysis of Special Detergents have also been completed and are published in Appendix II to this report as information. These methods will also be reviewed by the committee at its fall meeting. The committee has also been considering proposed specifications for built soap (powdered), and for trisodium phosphate, and these will be further discussed at the next meeting.

In view of the short time that the committee has been organized, it has made excellent progress and has cleared the way for much constructive work. The chairman wishes to record his appreciation for the splendid cooperation of all of the members in combating the tremendous difficulties which exist in some of the committee's problems.

RECOMMENDATIONS AFFECTING STANDARDS

Proposed Tentative Standards.—The committee submits for publication as tentative the following four specifications, one set of definitions and one method of chemical analysis, as appended hereto:

Proposed Tentative Specifications for:

Milled Toilet Soap,[1]
Caustic Soda,[1]
Modified Soda (Sesquicarbonate Type),[1] and
Soda Ash.[1]

Proposed Tentative Definitions of Terms Relating to Soaps and Detergents.[2]
Proposed Tentative Methods of Sampling and Chemical Analysis of Soaps and Soap Products[3]

These recommendations have been submitted to letter ballot of the committee which consists of 54 members, the results being as follows:

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[1] See pp. 1264, 1260, 1261 and 1262.
[2] See p. 1289.
[3] See p. 1266.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
PROPOSED TENTATIVE STANDARDS			
Specifications for Milled Toilet Soap	19	1	2
Specifications for Caustic Soda	20	0	2
Specifications for Modified Soda (Sesquicarbonate Type)	21	0	1
Specifications for Soda Ash	20	0	2
Methods of Sampling and Chemical Analysis of Soaps and Soap Products	14	0	1
Definitions of Terms Relating to Soaps and Detergents	20	1	1

This report has been submitted to letter ballot of the committee which consists of 54 members; 35 members returned their ballots, of whom 33 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

H. P. TREVITHICK,
Chairman.

B. S. VAN ZILE,
Secretary.

APPENDIX I

PROPOSED METHODS OF CHEMICAL ANALYSIS OF SULFONATED (SULFATED) OILS[1]

These are **proposed methods** and are published as information only. Comments are solicited and should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Scope

1. (*a*) These methods prescribe procedures for the chemical analysis of sulfonated (sulfated) oils.

(*b*) The analytical procedures appear in the following order:

		Section
Moisture	Method A. Water by Distillation with a Volatile Solvent	2 to 7
	Method B. Moisture and Volatile Matter by Hot-Plate Method	8 to 12
Organically Combined Sulfuric Anhydride	Method A. Titration Method	13 to 17
	Method B. Extraction-Titration Method	18 to 22
	Method C. Ash-Gravimetric Method (In the Presence of True Sulfonates)	23 to 26
Total De-Sulfated Fatty Matter		27 to 30
Total Active Ingredients		31 to 34
Unsaponifiable Non-Volatile Matter		35 to 39
Inorganic Salts		40 to 44
Total Alkalinity		45 to 47
Total Ammonia		48 to 50
Acidity as Free Fatty Acids	Method A. In the Absence of Ammonium or Triethanolamine Soaps or Both	51 to 53
	Method B. In the Presence of Dark Colored Oils but in the Absence of Ammonium or Triethanolamine Soaps or Both (Brine Method)	54 to 56
	Method C. In the Presence of Ammonium and/or Triethanolamine Soaps	57 to 59

MOISTURE

Method A. Water by Distillation with a Volatile Solvent

Scope

2. This method of analysis determines water existing in a sample of sulfonated (sulfated) oil by distilling the sample with a volatile solvent. The method is applicable only to sulfonated oils that do not contain the following: mineral acids, free sulfonic acids, or free sulfuric acid esters; or alkali hydroxides, carbonates or acetates; or alcohol, glycerol, diethylene glycol, acetone or other water-miscible volatile compounds.

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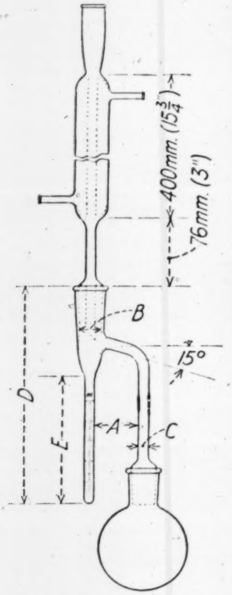
[1] Under the standardization procedure of the Society, these proposed methods are under the jurisdiction of the **A.S.T.M. Committee D-12 on Soaps and Detergents.**

Apparatus

3. The apparatus required consists of a glass flask, heated by suitable means and provided with a reflux condenser, discharging into a trap and connected to the flask. The connections between the trap and the condenser and flask shall be interchangeable ground joints. The trap serves to collect and measure the condensed water and to return the solvent to the flask.

(*a*) *Flask.*—A short-neck, round-bottom, glass flask (Fig. 1), made of well-annealed glass, and having an approximate capacity of 500 ml. shall be used.



A = 45 to 55 mm.
B = 22 to 24 mm., inside diameter
C = 9 to 11 mm., inside diameter
D = 235 to 240 mm.
E = 146 to 156 mm.

FIG. 1.—Apparatus for Determination of Water by Distillation with a Volatile Solvent, Method A.

(*b*) *Heat Source.*—The source of heat may be either an oil bath (stearic acid, paraffin wax, etc.) or an electric heater provided with a sliding rheostat, or other means of heat control.

(*c*) *Condenser.*—A water-cooled, reflux, glass-tube-type condenser (Fig. 1), having a jacket not less than 400 mm. (15$\frac{3}{4}$ in.) in length with an inner tube 9.5 to 12.7 mm. ($\frac{3}{8}$ to $\frac{1}{2}$ in.) in outside diameter shall be used. The end of the condenser to be inserted in the trap shall be ground off at an angle of 30 deg. from the vertical axis of the condenser and the tip of the condenser tube when inserted in the trap shall extend about 7 mm. ($\frac{1}{4}$ in.) above the surface of the liquid in the trap after the distillation conditions have been established.

(*d*) *Trap.*—A trap made of well-annealed glass constructed in accordance with Fig. 1 and graduated as shown from 0 to 5 ml. in 0.1-ml. divisions shall be used. The error of any indicated capacity may not be greater than 0.05 ml.

Special Solutions Required

4. (*a*) *Xylene, c.p.*

(*b*) *Oleic Acid.*—Oleic acid, U.S.P. grade, heated previous to use for 5 to 10 min. over a free flame at a temperature of 130 to 135 C.

Calibration

5. To calibrate the apparatus add approximately 1 g. of water to a mixture of 80 g. of xylene and 10 g. of oleic acid. Conduct the distillation as described in Section 6 (*b*), (*c*), and (*d*). When all the water has distilled, cool the apparatus, add another gram of water, and repeat the distillation. Continue the calibration up to the capacity of the receiving tube.

Procedure

6. (*a*) Before using, clean the condenser and the receiving tube thoroughly with soap and warm water, rinse well, then treat with hot cleaning solution (a mixture of 10 ml. of saturated potassium bichromate and 990 ml. of H_2SO_4 (sp. gr. 1.84)), and finally thoroughly wash and dry.

(*b*) Take enough of the sample to be tested for analysis to yield about 4 ml. of water. Introduce the approximate quantity into a weighing bottle and make the weighings from the bottle into the flask, taking care that after removal of the sample, no drops of oil are left on the outside of the weighing bottle. Add 80 g. of xylene, and oleic acid equivalent to about two and one-half times the weight of the bone-dry sample, to prevent foaming and jellying of the contents of the flask. Introduce glass beads to prevent bumping and mix the contents of the flask thoroughly by swirling, care being taken to avoid any loss of material. Fill the trap with xylene and immediately connect the flask with the distillation apparatus; insert a loose cotton plug in the top of the condenser tube to prevent condensation of atmospheric moisture in the condenser tube.

(*c*) Heat the flask and regulate the heating so that the condenser tube immediately below the water-jacket is just barely hot. In this way a minimum of water will condense further up the condenser, where it may be difficult to volatilize any moisture condensed on the walls.

(*d*) Continue the distillation at the specified rate until practically no water is visible on any part of the apparatus except within the graduations of the trap. This operation usually requires less than an hour. Increase the rate of distillation in order to remove all traces of condensed water in the condenser tube and continue the distillation until the water level in the trap remains unchanged after a 10-min. interval. Dislodge any droplets adhering to the side of the receiver with a thin copper wire twisted into a loop. Immerse the receiving tube in warm water at about 40 C. for 15 min., or until the xylene layer becomes clear, then read and record the temperature and the exact volume of the water in the trap.

TABLE I.—SPECIFIC GRAVITY OF WATER.[a]

TEMPERATURE, DEG. CENT.	SPECIFIC GRAVITY
+ 4	1.00000
35	0.99406
36	0.99371
37	0.99336
38	0.99299
39	0.99262
40	0.99224
41	0.99186
42	0.99147
43	0.99107
44	0.99066
45	0.99025

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[a] This table is taken from *Smithsonian Tables*, compiled from various authors.

Calculation

7. The volume of condensed water measured in the trap may be converted into its equivalent weight in grams by means of Table I. Calculate the percentage of water as follows:

$$\text{Water by weight, per cent} = \frac{\text{Grams of water}}{\text{Weight of sample in grams}} \times 100$$

Method B. Moisture and Volatile Matter by Hot-Plate Method

Scope

8. This method of analysis determines the percentage of water and other compounds volatile at about 100 C. existing in a sample of sulfonated (sulfated) oil by rapid evaporation. The method is applicable only to sulfonated oils that do not contain the following: mineral acids, free sulfonic acids or free sulfuric acid esters, ammonia, acetic acid or similar volatile acids, alkali hydroxides, carbonates, acetates or similar salts which may react with oleic acid at elevated temperatures liberating volatile acids, or glycerol, diethylene glycol, xylene or other compounds of similar volatility.

Apparatus

9. The apparatus required consists of a glass-stoppered weighing flask, a glass beaker, and a suitable thermometer.

(*a*) *Weighing Flask.*—Any suitable glass-stoppered weighing flask of 10 to 15-ml. capacity may be used.

(*b*) *Beaker.*—A Griffin low-form glass beaker with an approximate capacity of 150 ml. and a diameter of about 5 cm. shall be used.

(*c*) *Heat Source.*—The source of heat may be either an electric hot plate, with or without asbestos paper or board cover, or an open flame under a suitable asbestos board, and a wire gauze (to spread the heat).

(*d*) *Thermometer.*—A thermometer graduated from 90 to 150 C., about 3 in. in length, and substantially constructed shall be used.

Special Solutions Required

10. (*a*) *Oleic Acid, U.S.P.*

(*b*) *Desiccating Agent.*—Any suitable desiccating agent may be used.

NOTE.—Recent investigations seem to indicate that calcium chloride is unreliable as a laboratory desiccating agent.

Procedure

11. (*a*) Weigh approximately 5 g. of oleic acid, transfer it to the beaker and insert the thermometer. Heat the oleic acid gradually, while stirring with the thermometer, until the temperature reaches 130 C. Place the beaker in an oven at 105 to 110 C. for 15 min., cool in a desiccator, and weigh. Repeat the heating over the hot plate and in the oven until two successive weighings differ by less than 1.5 mg.

(*b*) Place about 6 g. of the sample in the weighing flask, and determine the weight accurately. Transfer the sample to the beaker (containing the thermometer), and weigh the flask again. Heat the mixture exactly as in the taring of the beaker as described in Paragraph (*a*). The loss in weight is equivalent to the moisture in the sample:

Calculation

12. Calculate the percentage of moisture and volatile matter in the sample as follows:

$$\begin{array}{c}\text{Moisture and volatile matter}\\ \text{(Hot-Plate Method), per cent}\end{array} = \frac{\text{Loss in weight of beaker in grams}}{\text{Weight of sample in grams}} \times 100$$

ORGANICALLY COMBINED SULFURIC ANHYDRIDE

Method A. Titration Method

Scope

13. This method of analysis determines the organically combined sulfuric anhydride existing in a sample of sulfonated (sulfated) oil by boiling the sample with sulfuric acid and determining the acidity of the reaction mixture. This method is applicable only to oils that split off their combined SO_3 upon boiling with mineral acids and that do not contain compounds which cannot be accurately titrated in water solution with methyl orange as the indicator.

Apparatus

14. The apparatus required consists of a glass flask provided with a glass stopper and an air condenser. The connection between the flask and the condenser shall be a ground joint. Perforated glass beads shall be used to prevent bumping.

(*a*) *Flask*.—An Erlenmeyer flask (Fig. 2) made of heat-resistant glass,[1] having a capacity of approximately 300 ml. and provided with a glass stopper shall be used.

(*b*) *Condenser*.—The condenser required consists of a glass tube, 915 mm. (36 in.) in length, and 8 mm. ($\frac{5}{16}$ in.) in outside diameter. The lower end of the tube shall be flared and ground to fit the mouth of the Erlenmeyer flask.

(*c*) *Glass Beads*.—Perforated glass beads, made of chemically-resistant glass, approximately 4 mm. ($\frac{5}{32}$ in.) in diameter shall be used. Before using, the glass beads shall be boiled thoroughly in several portions of water or until the wash-water reacts neutral to methyl orange indicator.

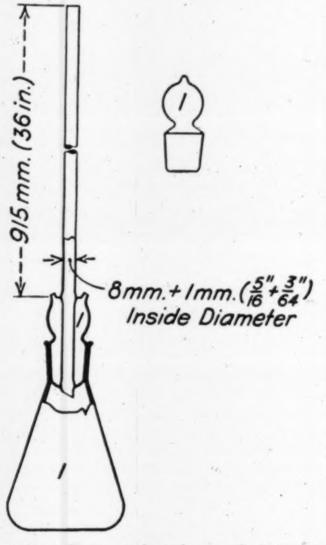

FIG. 2.—Apparatus for Determination of Organically Combined Sulfuric Anhydride, Method A.

The tolerance on the 8-mm. ($\frac{5}{16}$-in.) inside diameter tube is intended to be ± 1 mm. (± $\frac{3}{64}$ in.).

Special Solutions Required

15. (*a*) *Sodium Hydroxide* (*1 N*).—Accurately standardize a 1 *N* NaOH solution. Express the strength or concentration of the solution as milligrams of KOH per milliliter; 1 ml. of 1 *N* NaOH is equivalent to 56.1 mg. of KOH.

(*b*) *Sodium Hydroxide* (*0.5 N*).—Accurately standardize a 0.5 *N* NaOH solution. Express the strength of the solution as milligrams of KOH per milliliter; 1 ml. of 0.5 *N* NaOH is equivalent to 28.05 mg. of KOH.

(*c*) *Sulfuric Acid* (*Approximately 1 N*).

(*d*) *Sulfuric Acid* (*0.5 N*).—Accurately standardize a 0.5 *N* H_2SO_4

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[1] Pyrex glass is very satisfactory for this purpose.

solution. Express the strength of the solution as milligrams of KOH per milliliter; 1 ml. of 0.5 *N* H_2SO_4 is equivalent to 28.05 mg. of KOH.

(*e*) *Ethyl Ether, U.S.P.*

(*f*) *Sodium Chloride.*—Solid, c.p. NaCl.

(*g*) *Methyl Orange* (*0.1 per cent*).—Prepare a 0.1 per cent water solution of methyl orange indicator.

Procedure

16. The procedure consists of two determinations: namely, (*a*) the alkalinity of the sample, designated as *A*, and (*b*) the increase in acidity after boiling the sample with sulfuric acid, designated as *F*.

(*a*) *Alkalinity, A.*—Dissolve 10 g. of the sample in 100 ml. of water in the 250-ml. glass-stoppered Erlenmeyer flask, warming to obtain solution if necessary. After cooling, add 30 g. of NaCl, 25 ml. of ether, and five drops of methyl orange indicator, then add 0.5 *N* H_2SO_4, with frequent but gentle shaking until the mixture is slightly acid. Shake the contents of the flask vigorously, and complete the titration by adding first 0.5 *N* NaOH, several drops at a time, until the solution is alkaline, and then the acid, one or two drops at a time, until the endpoint is reached. Shake the solution vigorously after each addition of reagent. Drain burettes for 3 min. before readings are taken. Calculate the alkalinity, *A*, as follows:

$$A = \frac{(B \times D) - (C \times E)}{W}$$

where A = the total alkalinity in milligrams of KOH per gram,
B = the milliliters of acid,
C = the milliliters of alkali,
D = the strength of the acid in milligrams of KOH per milliliter,
E = the strength of the alkali in milligrams of KOH per milliliter, and
W = the weight of the sample in grams.

(*b*) *Increase in Acidity upon Boiling, F.*—Weigh 10 g. of the sample into the Erlenmeyer flask and boil for 1½ hr. under the air condenser with 1 *N* H_2SO_4 using glass beads to prevent bumping. The volume of H_2SO_4 added shall be sufficient to neutralize the total alkalinity, *A*, of the sample plus 25 ml. in excess. Regulate the heating so that the solution boils rather vigorously but with very little evaporation taking place. At the end of the heating period, allow the contents to cool, wash the condenser with a spray of water from a wash-bottle, disconnect the condenser. Add 30 g. of NaCl, 25 ml. of ether, 50 ml. of water and five drops of indicator, and titrate the solution with 1 *N* NaOH to the same endpoint as in the total alkalinity titration, Paragraph (*a*). During the titration stopper the flask frequently and shake the contents of the flask thoroughly. Drain the burettes for 3 min. before readings are taken.

NOTE.—Reserve the titrated solution for the subsequent determination of total de-sulfated fatty matter (Sections 27 to 30).

Make a blank determination simultaneously with the sample, using the same amount and strength of the H_2SO_4, approximately the same weight of glass beads, and heating and titrating under the same conditions as the sample.

Calculate the increase in acidity upon boiling, F, as follows:

$$F = \frac{(S - B)\,N}{W}$$

where F = the increase in acidity upon boiling in milligrams of KOH per gram,
S = the milliliters of NaOH necessary to titrate the sample,
B = the milliliters of NaOH necessary to titrate the blank,
N = the strength of the NaOH expressed as milligrams of KOH per milliliter, and
W = the weight of the sample in grams.

The increase in acidity, F, may be negative, in which case retain the sign obtained from the above formula in the calculation in Section 17.

Calculation

17. Calculate the percentage of organically combined sulfuric anhydride as follows:

$$\text{Organically combined sulfuric anhydride, per cent} = 0.1426\,(A + F)$$

where 0.1426 = one tenth of the molecular ratio of SO_3:KOH,
A = the total alkalinity in milligrams of KOH per gram, and
F = the increase in acidity upon boiling, expressed as milligram of KOH per gram. Retain the sign of F as obtained in Section 16 (*b*).

Method B. Extraction-Titration Method

Scope

18. This method of analysis determines the organically combined sulfuric anhydride existing in a sample of sulfated oil by extracting the undecomposed sulfonated or sulfated fat and other fatty matter over an acidulated, concentrated salt solution, boiling the residue with sulfuric acid after evaporating the solvent, and titrating the products of reaction. This method is applicable only to sulfated oils that split off their combined SO_3 upon boiling with mineral acids, including samples containing sodium acetate or other compounds that cannot be accurately titrated in water solution with methyl orange as the indicator.

Apparatus

19. The apparatus shall be the same as described for Method *A* in Section 14.

Special Solutions Required

20. The solutions shall be the same as described for Method *A* in Section 15.

Procedure

21. The procedure consists of isolating and purifying the fatty matter as it exists in the original oil by dissolving the sample in a solvent, acidifying and washing with saturated brine, and determining the increase in acidity upon boiling the isolated product with sulfuric acid. This increase in acidity is designated as *F*.

(*a*) *Separation of Purified Oil.*—Weigh 5 to 10 g. of the sample, depending upon the concentration of the fatty matter, into a 250-ml. separatory funnel containing 50 ml. of concentrated NaCl solution, some solid NaCl, 5 drops of methyl orange indicator, and 50 ml. of ether. Shake the mixture and neutralize with approximately 1 *N* H_2SO_4 until the lower layer is distinctly pink (about 0.2 ml. in excess).

NOTE.—Highly sulfonated oils at this stage may form three layers instead of two. In such cases, use a fat solvent consisting of a mixture of two parts of ether and one of alcohol.

Allow the mixture in the separatory funnel to settle for at least 5 min., draw off the lower layer into another separatory funnel and wash the ether layer with 25-ml. portions of NaCl solution until practically neutral to methyl orange, that is, until 1 drop of 0.5 *N* NaOH turns the wash water strongly alkaline. Allow all separations to settle for at least 5 min. Combine the water layers, and extract with two 25-ml. portions of ether. Combine the last two ether extractions and wash with NaCl solution until free from acid, as in the case of the ether layer in the first funnel. Combine all the ether layers in the decomposition flask and evaporate the ether.

(*b*) *Increase in Acidity Upon Boiling, F.*—Determine the increase in acidity upon boiling in accordance with the procedure described for Method *A* in Section 16 (*b*). (See Note.)

NOTE.—Reserve the titrated solution for the subsequent determination of total de-sulfated fatty matter (Sections 27 to 30).

Make a blank determination as described for Method *A* in Section 16 (*b*).

Calculate the increase in acidity *F* in accordance with Section 16 (*b*).

Calculation

22. Calculate the percentage of organically combined sulfuric anhydride as follows:

$$\begin{array}{l}\text{Organically combined sulfuric anhydride} \\ \text{(Extraction-titration method), per cent}\end{array} = 0.1426 \times F$$

where 0.1426 = one tenth the molecular ratio of SO_3: KOH, and
F = the increase in acidity upon boiling.

Method C. Ash-Gravimetric Method (In the Presence of True Sulfonates)

Scope

23. This method of analysis determines the organically combined sulfuric anhydride existing in a sample of sulfonated oil by extracting the undecomposed sulfonated or sulfated fat and other fatty matter over an acidulated, concentrated salt solution, and ashing the purified extract. This method is applicable to all types of sulfonated oils, including true sulfonic acid oils and those containing sodium acetate or similar partially titratable compounds.

Special Solutions Required

24. (*a*) *Ethyl Ether, U.S.P.*

(*b*) *Sodium Chloride.*—Solid, c.p. NaCl.

(*c*) *Sodium Sulfate.*—Anhydrous, c.p. Na_2SO_4.

(*d*) *Methyl Orange (0.1 per cent).*—Prepare a 0.1 per cent water solution of methyl orange indicator.

Procedure

25. The procedure consists of isolating and purifying the fatty matter as it exists in the original oil by dissolving the sample in a solvent, acidifying and washing with saturated brine, and ashing the purified extract. If the sample contains ammonia, the ammonia shall first be expelled before the determination is made on the sample.

(*a*) *In the Absence of Ammonia.*—Proceed as described in the separation of purified oil (Section 21 (*a*)), combining the ether layers in the first funnel rather than in the decomposition flask. Carefully remove any water that may settle and dehydrate the ether layer as follows: add 5 g. of anhydrous Na_2SO_4, shake vigorously for 5 min., and filter directly into a 150-ml. beaker, placed in a bath of warm water. Wash the flask and filter with ether until free from fat (absence of oil stains on the filter paper after drying) and add the filtrate to the beaker. To avoid creeping of the oil, the volume in the beaker should at no time during the filtering and washing exceed 50 ml. (one-third full).

Evaporate the ether solution until the volume has been reduced to about 20 ml. and transfer the residue to a tared 50-ml. crucible (high form). Immerse the crucible in a 100-ml. beaker containing warm water until practically all of the ether has evaporated. Rinse the beaker with two 10-ml. and three 5-ml. portions of ether, respectively, or until all of the oil has been transferred to the crucible; allow each portion of rinsing ether to evaporate before the next rinsing is made. Burn gently the solvent-free residue, and finally ignite at a dull red heat to constant weight.

To prevent creeping of the oil and to hasten evaporation, stir the solution with a glass rod; before the oil is burned, wipe the rod clean with ashless filter paper and add the paper to the crucible. To oxidize traces of carbon or sodium sulfide that might form, moisten the ash with hydrogen peroxide (30 per cent) and again carefully ignite to constant weight. Calculate the percentage of ash as follows:

$$\text{Extracted ash, per cent} = \frac{\text{Weight of ash in grams}}{\text{Weight of sample in grams}} \times 100$$

(*b*) *In the Presence of Ammonia.*—Dissolve 5 to 8 g. of the sample in 80 ml. of water in a 300-ml. beaker. Add 10 ml. of 1 *N* NaOH and boil the solution gently until wet litmus paper no longer indicates ammonia. Cool the solution and transfer to a 300-ml. separatory funnel and add about 35 g. of solid NaCl or enough to make the final solution a 25 per cent NaCl solution. Add 5 drops of methyl orange and complete the neutralization, extraction, etc., in accordance with the procedure described in Paragraph (*a*).

Calculation

26. Calculate the percentage of organically combined sulfuric anhydride as follows:

$$\text{Organically combined sulfuric anhydride, per cent} = 1.1267 \times \text{percentage of the extracted ash}$$

where 1.1267 = the molecular ratio of $2SO_3:Na_2SO_4$.

TOTAL DE-SULFATED FATTY MATTER

Scope

27. This method of analysis determines the total de-sulfated fatty matter in a sample of sulfated oil by decomposition with diluted mineral acids and extraction of the decomposed fat. This method is not applicable to samples that are not completely decomposed upon boiling with mineral acids.

Special Solutions Required

28. (*a*) *Sulfuric Acid (Approximately 1 N).*

(*b*) *Ethyl Ether, U.S.P.*

Procedure

29. The procedure consists in decomposing the sample with sulfuric acid, extracting the fatty matter with ether, evaporating the solvent, and weighing the residue. After cooling, transfer the titrated solution obtained after determining organically combined sulfuric anhydride, in accordance with Method *A* or Method *B* (Sections 16 (*b*) or 21 (*b*)), into a 250-ml. separatory funnel and shake with 50 ml. of ether. Draw off the water

layer into another separatory funnel and extract twice with 25-ml. portions of ether. Wash the combined ether layers with 15-ml. portions of water until the wash water is neutral to methyl orange. Transfer the ether layer to a tared 150-ml. beaker, evaporate on the water bath until practically free from solvent, dry in a hot-air oven at 105 to 110 C. for 30 min., cool in a desiccator and weigh. Repeat the heating for 30-min. periods until constant weight is obtained.

NOTE.—Reserve the extracted fatty matter for the subsequent determination of unsaponifiable matter (Sections 35 to 39).

Calculation

30. Calculate the total de-sulfated fatty matter as follows:

$$\text{Total de-sulfated fatty matter, per cent} = \frac{\text{Weight of residue in grams}}{\text{Weight of sample in grams}} \times 100$$

TOTAL ACTIVE INGREDIENTS

Scope

31. This method of analysis determines the total active ingredients in a sample of sulfonated or sulfated oil as it exists in the original sample by extracting the undecomposed sulfonated or sulfated fat and other fatty matter over an acidified concentrated salt solution. Free alkali or alkali bound as soap is not included.

NOTE.—In the case of sulfated oils only, this determination may also be estimated by calculation (see Section 34 (*c*)) as it is equivalent to the sum of the de-sulfated fatty matter and neutralized organically combined sulfuric anhydride.

Special Solutions Required

32. (*a*) *Ethyl Ether, U.S.P.*

(*b*) *Sodium Chloride.*—Solid, c.p. NaCl.

(*c*) *Sodium Sulfate.*—Anhydrous, c.p. Na_2SO_4.

(*d*) *Methyl Orange* (*0.1 per cent*).—Prepare a 0.1 per cent water solution of methyl orange indicator.

(*e*) *Alcoholic Potassium Hydroxide* (0.5 *N*).—Accurately standardize a 0.5 *N* alcoholic KOH solution the strength of which shall be expressed as milligrams of KOH per milliliter; 1 ml. of 0.5 *N* alcoholic KOH is equivalent to 28.05 mg. of KOH.

Procedure

33. (*a*) The procedure consists of isolating and purifying the fatty matter as it exists in the original oil by dissolving the sample in a solvent, acidifying and washing with saturated brine, and weighing the purified extract. Proceed as described in the determination of organically combined sulfuric anhydride, Method *C*, in the absence of ammonia (Section 25 (*a*)), retaining the separated active ingredients in the 150-ml. beaker instead of transferring it to a crucible. Evaporate the filtrate to a total

volume of about 20 ml., add exactly 2 ml. of alcoholic KOH, mix the contents by swirling, and finally evaporate until practically free from ether.

NOTE.—With highly sulfonated oil, it may be necessary to add 5 to 10 ml. of the alcoholic KOH in order to stabilize the residue.

(*b*) Dry the residue at 108 to 112 C. for $1\frac{1}{2}$ hr., cool in a desiccator, and weigh. Repeat the heating for 30-min. periods until constant weight is obtained.

Calculation

34. (*a*) *Correction for the Alkali Added.*—Calculate the correction for the alkali added as follows:

$$\text{Correction for alkali added, grams} = 0.0006791\ (\text{milliliters of KOH} \times S)$$

where 0.0006791 = the molecular ratio of (K − H) : KOH divided by 1000, and
S = the strength of the KOH in milligrams of KOH per milliliter.

(*b*) *Total Active Ingredients.*—Calculate the total active ingredients as follows:

$$\text{Total active ingredients, per cent} = \frac{\text{Weight of residue in grams} - \text{alkali correction}}{\text{Weight of sample in grams}} \times 100$$

(*c*) *Total Active Ingredients Calculation Method* (*Applicable to Sulfated Oils only*).—Calculate the total active ingredients by the calculation method which is applicable to sulfated oils only, as follows:

$$\begin{matrix}\text{Total active}\\ \text{ingredients, per cent}\end{matrix} = \begin{matrix}\text{Percentage of total}\\ \text{de-sulfated fatty matter}\end{matrix} + \left(\frac{NaSO_4-}{SO_3} \times \begin{matrix}\text{percentage of}\\ \text{combined } SO_3\end{matrix}\right)$$

or

$$\begin{matrix}\text{Total active}\\ \text{ingredients, per cent}\end{matrix} = \begin{matrix}\text{Percentage of total}\\ \text{de-sulfated fatty matter}\end{matrix} + 1.4875 \times \begin{matrix}\text{percentage of}\\ \text{combined } SO_3\end{matrix}$$

NOTE.—The factor 1.4875 is based on the assumption that the de-sulfated fatty matter becomes polymerized at the point where the SO_3 splits off and no hydroxyl groups are formed. If the latter are formed the factor becomes $\frac{NaSO_3 - 1}{SO_3} = 1.275$. Calculated results were in better agreement with actual determinations when the higher factor was used.

UNSAPONIFIABLE NON-VOLATILE MATTER

Scope

35. This method of analysis determines the unsaponifiable, non-volatile (above 80 C.) matter existing in a sample of sulfated oil by saponifying the de-sulfated fatty matter and extracting the unsaponifiable matter from the soap solution with ethyl ether.

Apparatus

36. *Saponification Flask.*—The apparatus required for the saponification consists of a glass flask provided with an air condenser and identical with the apparatus described in Method *A* for organically combined sulfuric anhydride (Section 14 (*a*) and (*b*)).

Special Solutions Required

37. (*a*) *Potassium Hydroxide Solution* (*0.5 N*).—Prepare an approximately 0.5 *N* aqueous KOH solution.

(*b*) *Alcoholic Potassium Hydroxide* (*0.5 N*).—Prepare an approximately but not less than 0.5 *N* alcoholic KOH solution.

(*c*) *Ethyl Ether, U.S.P.*

Procedure

38. The procedure consists in decomposing the sample with mineral acid, extracting the de-sulfated fatty matter, saponifying the latter, and extracting the unsaponifiable matter from the soap solution with ethyl ether.

(*a*) *De-Sulfated Fatty Matter.*—Determine the de-sulfated fatty matter as described in the determination of total de-sulfated fatty matter (Section 29).

(*b*) *Saponification.*—Accurately weigh 2 to 2.5 g. of the de-sulfated fatty matter in the flask, add 25 ml. of the alcoholic KOH and simmer the contents (without loss of alcohol) for 1 hr. with occasional swirling, over an electric hot plate or other source of heat.

(*c*) *Extraction.*—Transfer the contents of the flask to a 250-ml. separatory funnel and wash the flask several times with a total of 50 ml. of water, pouring it into the separatory funnel. Extract the solution, while still warm (about 30 C.), with 50 ml. of ether (rinse the saponification flask with the ether before adding it to the separatory funnel), shaking vigorously for about 1 min., and allow the layers to settle and clear. Draw off the lower layer in a second 250-ml. separatory funnel and extract in a similar manner with two 50-ml. portions of ether.

(*d*) *Preliminary Water Washing.*—Add 20 ml. of water to the combined ether layer, turn the separatory funnel and contents over gently about six times, and allow the layers to settle and clear. Draw off the lower layer and discard. Wash the ether layer twice more with 20-ml. portions of water, shaking vigorously after each addition.

(*e*) *Alkali and Water Washing.*—Add 20 ml. of 0.5 *N* KOH to the washed ether layer, turn the separatory funnel and contents over gently about six times, and allow the layers to settle and clear. Draw off the lower layer and discard. Wash the ether layer with 20 ml. of water, shaking vigorously. Continue washing with 20 ml. of 0.5 *N* KOH followed by 20 ml. of water, shaking vigorously after each addition, until the alkali layer upon strong acidification with HCl and settling for a few minutes is only faintly opalescent. Finally wash the ether layer with 20-ml. portions of water until the wash water is no longer pink to phenolphthalein.

(*f*) *Solvent Removal.*—Transfer the ether layer to a 150-ml. beaker (counter-balance the beaker if the amount of unsaponifiable matter is small and important) and evaporate over a hot-water bath until practically free of ether. Heat the beaker at 75 to 80 C. until constant weight is obtained.

NOTE 1.—A 30 per cent alcoholic solution by volume is the most satisfactory solution from which to extract the unsaponifiable matter.

NOTE 2.—If emulsions form, add 5 ml. of alcohol, pouring it down the side of the funnel.

NOTE 3.—It is important to maintain the volume of ether at not less than 150 ml., otherwise small quantities of unsaponifiable matter may be lost.

NOTE 4.—To check whether or not the unsaponifiable matter is free from fatty acids, dissolve the residue in 10 ml. of freshly boiled neutral alcohol and titrate with 0.1 *N* alcoholic KOH solution, using phenolphthalein as the indicator. Not more than 0.1 ml. should be required for neutralization. If more is required, the determination has not been carried out effectively and must be repeated.

Calculation

39. Calculate the unsaponifiable matter as follows:

$$\text{Unsaponifiable non-volatile matter, percent} = \frac{\text{Weight of residue in grams}}{\text{Weight of de-sulfated fatty matter in grams}} \times \text{percentage of de-sulfated fatty matter}$$

INORGANIC SALTS

Scope

40. This method of analysis determines in a sample of sulfonated oil the inorganic sulfates, chlorides, and all other salts that are insoluble in a mixture of oleic acid and carbon tetrachloride. The method consists in dehydrating the sample, dissolving in a solvent, filtering and weighing the residue. The presence of sodium acetate does not interfere with this method.

Apparatus

41. Either a Gooch crucible or filter paper may be used for filtering. Ignite the Gooch crucible in a larger crucible, supported by an asbestos ring and assembled as shown in Fig. 3. If filter paper is used, it may be a 9-cm. Whatman No. 40 or similar grade of ashless filter paper.

Special Solutions Required

42. (*a*) *Oleic Acid, U.S.P.*

(*b*) *Carbon Tetrachloride, U.S.P.*

(*c*) *Ethyl Ether, U.S.P.*

(*d*) *Asbestos.*—Asbestos fiber, alkali and acid washed.

Procedure

43. The procedure consists of dehydrating the sample, dissolving in a solvent, filtering, and igniting the residue. In the presence of ammonium salts, the residue is not ignited but only dried to constant weight.

(*a*) *In the Absence of Ammonium Salts.*—Weigh 3 to 5 g. of the sample and place in a 250-ml. beaker, add an approximately equal amount of oleic acid, and heat the mixture on an oil bath, while stirring constantly with a thermometer, at a temperature of 105 to 110 C. until practically free from water. Continue the heating until the temperature of the contents reaches 118 to 120 C., and maintain at that temperature for about 5 min. If the dehydrated sample upon cooling does not remain liquid, add more oleic acid. Dissolve the dehydrated sample in 100 ml. of CCl_4 warmed to 50 to 55 C., and filter through a counterpoised filter paper or a Gooch crucible.

Prepare the Gooch crucible by passing a thin emulsion of asbestos fiber in water through the crucible until a pad of about 2 mm. in thickness is formed. Dry the asbestos pad and ignite for 30 min. at first gently and finally more strongly. Then pass 75 ml. of CCl_4 through the crucible and again ignite the asbestos pad, cool in a desiccator, and weigh. Repeat the process of washing with CCl_4 until there is no further loss in weight.

Wash the residue with three 15-ml. portions of a solution of oleic acid in CCl_4 (2 per cent), then with six 15-ml. portions of hot CCl_4, and finally with two 15-ml. portions of ether or until the residue is free from oil. Take care that the top of the filter is thoroughly washed. Transfer the last traces of the residue to the filter by allowing the solvent to evaporate when the salts become free flowing. Dry the residue at 125 to 130 C. for 45 min., cool in a desiccator, and weigh. Ignite the residue at a dull red heat for 15 min., weigh, and repeat the ignition until constant weight is obtained.

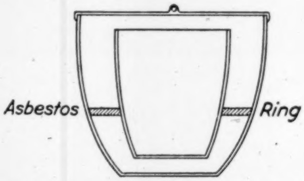

FIG. 3.—Gooch Crucible Assembly for Determination of Inorganic Salts.

(*b*) *In the Presence of Ammonium Salts.*—Proceed as described in Paragraph (*a*) for the determination of inorganic salts in the absence of ammonium salts with the following exceptions: (*1*) in preparing the Gooch crucible, do not ignite but heat it at 125 to 130 C. for 45 min., and repeat the heating until constant weight is obtained, and (*2*) heat the residue, whether in a Gooch crucible or on a filter paper as in (*1*), but do not ignite it.

Calculation

44. The method of calculation depends upon whether or not ammonium salts are present in the sample.

(*a*) Calculate the inorganic sulfates and chlorides including ammonium salts as follows:

$$\text{Inorganic sulfates and chlorides, including ammonium salts, per cent} = \frac{\text{Weight of dried residue in grams}}{\text{Weight of sample in grams}} \times 100$$

NOTE.—In the absence of ammonium salts, the difference between the percentage of the dried residue and the percentage of the ignited residue shall not be greater than 0.25 per cent.

(*b*) Calculate the non-volatile inorganic sulfates and chlorides (in the absence of ammonium salts) as follows:

$$\text{Non-volatile, inorganic sulfates and chlorides, per cent} = \frac{\text{Weight of ignited residue in grams}}{\text{Weight of sample in grams}} \times 100$$

TOTAL ALKALINITY

Scope

45. This method of analysis determines the total alkalinity existing in a sample of sulfonated and sulfated oil by titrating a water solution of the sample with mineral acid in the presence of methyl orange as the indicator. This method determines the total alkalinity of the fixed alkali, ammonia and triethanolamine bound as soap, free alkali, and the alkalinity of titratable alkaline salts, but not that of non-titratable alkaline salts.

Procedure

46. Proceed as described in the determination of alkalinity under organically combined sulfuric anhydride, Method *A* (Section 16 (*a*)).

Calculation

47. Calculate the total alkalinity as described in Section 16 (*a*)).

TOTAL AMMONIA

Scope

48. This method of analysis determines the total ammonia in a sample of sulfonated (sulfated) oil by boiling a water solution of the sample with excess alkali and determining by titration the loss in alkali after the boiling.

Procedure

49. The procedure consists of two determinations: namely, (*a*) the total alkalinity, and (*b*) the loss in alkalinity after boiling with excess NaOH.

(*a*) *Total Alkalinity.*—Determine the total alkalinity as described in the determination of organically combined sulfuric anhydride, Method *A* (Section 16 (*a*)).

(*b*) *Alkalinity After Boiling.*—Dissolve 10 g. of the sample in 100 ml. of water in a 500-ml. beaker, add 25 ml. of 0.5 *N* NaOH and boil the mixture for 30 min. or until all the ammonia is expelled as indicated by moistened red litmus paper. Cool the contents of the beaker, add methyl orange indicator (0.1 per cent) and titrate to an approximate endpoint. Transfer the mixture to a 250-ml. glass-stoppered flask and complete the titration (with the addition of salt and ether) as described in the determination of organically combined sulfuric anhydride, Method *A* (Section 16 (*b*)).

Calculation

50 Calculate the total ammonia as follows:

$$\text{Total ammonia, milligrams of KOH per gram} = A + \left(\frac{(B \times D) - (C \times E)}{W}\right)$$

or

$$\text{Total ammonia, per cent} = 0.0303\ (\text{total ammonia as milligrams of KOH})$$

where A = the total alkalinity in milligrams of KOH per gram,
B = the milliliters of alkali added,
C = the milliliters of acid added,
D = the strength of the alkali as milligrams of KOH per milliliter,
E = the strength of the acid as milligrams of KOH per milliliter, and
W = the weight of the sample in grams.

ACIDITY AS FREE FATTY ACIDS

Method A. In the Absence of Ammonium or Triethanolamine Soaps or Both

Scope

51. This method of analysis determines the acidity as free fatty acids existing in a sample of sulfonated and sulfated oil by titrating the sample dissolved in a solvent. It is not applicable in the presence of ammonium or triethanolamine soaps or salts, or other compounds which do not react neutral to phenolphthalein when dissolved in alcohol.

Procedure

52. The procedure consists of titrating a solution of the sample in a mixture of alcohol and ether in the presence of phenolphthalein indicator. Dissolve 10 g. of the sample in a mixture of 50 ml. of neutral alcohol and 25 ml. of ether, add five drops of phenolphthalein indicator, and titrate the solution with 0.5 N NaOH or KOH until the pink color persists after vigorous shaking.

Calculation

53. Calculate the acidity as free fatty acids, as follows:

$$\begin{array}{l}\text{Acidity as free fatty acids,}\\ \text{milligrams of KOH per}\\ \text{gram, calculated as acid}\\ \text{number}\end{array} = \frac{\text{Milliliters of alkali} \times \text{strength as milligrams of KOH}}{\text{Weight of sample in grams}}$$

Method B. In the Presence of Dark Colored Oils but in the Absence of Ammonium or Triethanolamine Soaps or Both (Brine Method)

Scope

54. This method of analysis determines the acidity as free fatty acids existing in a sample of sulfonated (sulfated) oil by titrating a water solution of the sample with phenolphthalein as the indicator. It is not applicable in the presence of ammonia or triethanolamine soaps or acid salts.

Procedure

55. The procedure consists in titrating a water solution of the sample in the presence of salt, ether, and alcohol with phenolphthalein as the indicator. Dissolve 10 g. of the sample in 100 ml. of water contained in a 250-ml., glass-stoppered, Erlenmeyer flask, warming to obtain solution, if necessary. After cooling, add 30 g. of NaCl, 25 ml. of ether and 50 ml. of neutral alcohol.

NOTE.—At this stage, methyl orange indicator may be added and the total alkalinity determined as described in the determination of organically combined sulfuric anhydride, Method *A* (Section 16 (*a*)).

Add five drops of phenolphthalein indicator (1.0 per cent alcoholic solution) and titrate the contents of the flask with 0.5 *N* NaOH until the first pink tinge appears in the water layer, shaking vigorously after each addition of the reagent.

Calculation

56. Calculate the acidity as free fatty acids as described in the determination of acidity as free fatty acids, Method *A* (Section 53).

NOTE.—In case the total alkalinity was determined on the same sample the free fatty acids is calculated as follows:

$$\text{Acidity as free fatty acids, milligrams KOH per gram} = \frac{\text{Milliliters of alkali} \times \text{strength as milligrams of KOH}}{\text{Weight of sample in grams}} - \left(\begin{array}{c}\text{total}\\ \text{alka-}\\ \text{linity}\end{array}\right)$$

Method C. In the Presence of Ammonium and/or Triethanolamine Soaps

Scope

57. This method of analysis determines the acidity as free fatty acids in the presence of ammonium and/or triethanolamine soaps, existing in a sample of sulfated or sulfonated oil or both, by calculation from the total alkalinity and the free-and-alkali-combined fatty acids.

Procedure

58. The procedure consists of two determinations: namely, (*a*) total alkalinity, and (*b*) free-and-alkali-combined fatty acids.

(*a*) *Total Alkalinity.*—Determine the total alkalinity as described in the determination of organically combined sulfuric anhydride, Method *A* (Section 16 (*a*)).

(*b*) *Free-and-Alkali-Combined Fatty Acids.*—Extract the total sulfated and sulfonated fatty matter as described in the determination of total active ingredients (Section 33 (*a*) and (*b*)), but omit the dehydration of the extract.

NOTE.–In the presence of acetates, wash the ether layer carefully with concentrated NaCl solution until one or two drops of 0.5 *N* NaOH added to the final wash water are neutral to phenolphthalein indicator.

Add exactly 0.5 ml. of 0.5 *N* NaOH to the extract, evaporate the ether to a volume of about 25 ml., add approximately 50 ml. of neutral alcohol, and titrate the mixture with 0.5 *N* NaOH.

Calculation

59. Calculate the free-and-alkali-combined fatty acids as described in the determination of acidity as free fatty acids, Method *A* (Section 53). Calculate the acidity as free fatty acids in the presence of ammonium and/or triethanolamine soaps, as follows:

$$\begin{array}{c}\text{Acidity as free fatty acids,}\\ \text{milligrams of KOH per gram}\end{array} = (\text{Free-and-alkali-combined fatty acids}) - \begin{array}{c}\text{(total alka-}\\ \text{linity).}\end{array}$$

APPENDIX II

PROPOSED METHODS OF CHEMICAL ANALYSIS OF SPECIAL DETERGENTS[1]

These are **proposed methods** and are published as information only. Comments are solicited and should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Scope

1. These methods cover procedures for the chemical analysis of inorganic alkaline detergents.

CAUSTIC SODA

Sampling

2. (*a*) *Flake Caustic Soda.*—Flake caustic soda shall be sampled by removing portions from various parts of the drum.

(*b*) *Powdered Caustic Soda.*—Powdered caustic soda shall be sampled by inserting a sampling tube through the contents of the drum in several places. The tube shall be dried by heating just before use.

(*c*) *Fused Caustic Soda.*—Fused caustic soda shall be sampled by taking chipped samples from the center and bottom of the drum and then mixing the gross sample in the approximate proportions in which the tops and bottoms occur in the drum.

(*d*) *Precautions.*—Caustic soda shall not be sampled in a moist atmosphere. In the case of fused caustic soda the portion taken for analysis shall have the surface layer of carbonate scraped off immediately before transferring to the weighing bottle. In all cases the sample shall be transferred to a thoroughly dried weighing bottle immediately after it is taken; the bottle shall be tightly stoppered at once.

Total Alkalinity as Na_2O

3. Weigh 10 g. of the sample, dissolve in CO_2-free distilled water, wash into a 500-ml. volumetric flask and dilute to volume with CO_2-free distilled water. Protect the solution from the air as much as possible. Pipette a one-fifth aliquot into a 400-ml. beaker and determine Na_2O by titrating the sample against 1.0 N acid using methyl red as the indicator. Calculate the total alkalinity as Na_2O, as follows:

$$Na_2O\text{, per cent} = \frac{A \times 5 \times 3.1}{\text{Weight of sample in grams}}$$

where A = the milliliters of acid necessary to titrate the Na_2O in the sample.

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[1] Under the standardization procedure of the Society these proposed methods are under the jurisdiction of the A.S.T.M. Committee D-12 on Soaps and Detergents.

Sodium Hydroxide

4. Determine the NaOH on a second one-fifth aliquot which is pipetted into a 250-ml. Erlenmeyer flask. Add about 25 ml. of neutral $BaCl_2$ solution (10 per cent) and titrate the sample with 1.0 *N* acid using phenolphthalein as the indicator. Calculate the percentage of NaOH as follows:

$$\text{NaOH, per cent} = \frac{B \times 5 \times 4.0}{\text{Weight of sample in grams}}$$

where B = The milliliters of acid necessary to titrate the NaOH in the sample.

Carbonate as Na_2CO_3

5. Calculate the carbonate as Na_2CO_3, as follows:

$$Na_2CO_3\text{, per cent} = \frac{(A - B) \times 5 \times 5.3}{\text{Weight of sample in grams}}$$

where A = the milliliters of acid necessary to titrate the Na_2O in the sample, and

B = the milliliters of acid necessary to titrate the NaOH in the sample.

SODA ASH

Sampling

6. Soda ash shall be sampled by removing portions from various parts of the container. Samples shall not be taken from those portions of the soda ash where caking is noticeable due to the absorption of moisture and CO_2 through the container. If the soda ash is caked, the sample shall be obtained by thoroughly mixing and quartering the entire contents of the package.

Moisture

7. Determine the moisture content by drying 2.0 g. of the sample at 150 to 155 C. for 1 hr.

Total Alkalinity as Na_2CO_3

8. Analyses for carbonate and bicarbonate shall be referred to the dry basis or carried out on samples previously dried as described in Section 7. Weigh 5.3 g. of the sample and transfer to a 500-ml. Erlenmeyer flask. Dissolve the sample in 100 ml. of distilled water, add exactly 100 ml. of 1.0 *N* H_2SO_4 from a standard burette, place a small funnel in the neck of the flask, and boil gently for 5 min. to expel CO_2. Cool, rinse down the sides of the flask, add 4 drops of methyl red indicator and titrate with 0.1 *N* NaOH. Calculate the total alkalinity as Na_2CO_3, as follows:

$$\text{Total alkalinity as } Na_2CO_3\text{, per cent} = \text{Milliliters of acid} - \frac{\text{Milliliters of NaOH}}{10}$$

Sodium Bicarbonate

9. Weigh 8.4 g. of the sample and transfer to a 250-ml. beaker, dissolve in 100 ml. of distilled water, and titrate with 1.0 N NaOH until a drop of the solution added to a drop of freshly prepared $AgNO_3$ solution (10 per cent) on a spot plate gives a dark color instantly. Calculate the percentage of sodium bicarbonate as follows:

$$NaHCO_3\text{, per cent} = \text{Milliliters of 1.0 } N \text{ NaOH}$$

Calculate the percentage of Na_2CO_3 as follows:

$$Na_2CO_3\text{, per cent} = \text{Total alkalinity as } Na_2CO_3 \text{ in per cent} - (\text{percentage of } NaHCO_3 \times 0.6309)$$

Matter Insoluble in Water

10. Dissolve 20 g. of the sample in 300 ml. of distilled water in a 400-ml. beaker. Filter through a previously prepared, dried and weighed Gooch crucible. Wash the residue free from alkali with distilled water and dry in an oven at 100 C. Calculate the percentage of matter insoluble in water as follows:

$$\text{Matter insoluble in water, per cent} = \text{Weight of residue} \times 5$$

Apparent Density

11. Weigh 30 g. of the sample and transfer to a 100-ml. graduate. Rotate the graduate until the sample flows freely and then, taking great care to avoid jarring, level the surface of the sample, and read the volume. Calculate the apparent density as follows:

$$\text{Apparent specific gravity} = \frac{30}{\text{Volume in milliliters}}$$

$$\text{Apparent density, lb. per cu. ft.} = \text{Apparent specific gravity} \times 62.5$$

MODIFIED SODA (SESQUI-CARBONATE TYPE)

Sampling

12. The sample of modified soda (sesqui-carbonate type) shall be selected as described in Section 6 for the sampling of soda ash.

Total Alkalinity as Na_2O

13. Weigh 3.1 g. of the sample and dissolve in about 100 ml. of distilled water in a 500-ml. Erlenmeyer flask. Add 4 drops of methyl red indicator, and enough 1.0 N H_2SO_4 to reach the endpoint plus approximately 1 ml. in excess. Place a small funnel in the neck of the flask and boil for 5 min. to expel CO_2. The solution should still be acid after boiling. Rinse

down the sides of the flask and titrate back with 0.1 *N* NaOH. Calculate the total alkalinity as Na_2O, as follows:

$$\text{Total alkalinity as } Na_2O\text{, per cent} = \text{Milliliters of acid} - \frac{\text{Milliliters of NaOH}}{10}$$

Sodium Bicarbonate and Sodium Carbonate

14. (*a*) Weigh 8.4 g. of the sample and dissolve in about 100 ml. of distilled water in a 250-ml. beaker. Titrate the sample with 1.0 *N* NaOH until a drop of the solution added to a drop of freshly prepared $AgNO_3$ solution (10 per cent) on a spot plate gives a dark color instantly. Calculate the sodium bicarbonate as follows:

$$NaHCO_3\text{, per cent} = \text{Milliliters of } 1.0\ N\ \text{NaOH}$$

(*b*) *Sodium Carbonate*.—Calculate the percentage of sodium carbonate as follows:

$$Na_2CO_3\text{, per cent} = (\text{Percentage of } Na_2O - \text{percentage of } NaHCO_3 \times 0.3690) \times 1.7097$$

Matter Insoluble in Water

15. Determine the matter insoluble in water in accordance with the procedure in Section 10.

PHOSPHATES, SILICATES AND BORATES

Phosphates, Silicates, and Borates[1]

16. Determine these alkaline salts in accordance with the analytical procedures described in the Tentative Methods of Sampling and Chemical Analysis of Soaps and Soap Products (A.S.T.M. Designation: D 460 – 37 T) of the American Society for Testing Materials.[2]

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[1] The committee has in mind preparing more complete analytical procedures for these alkaline salts when the demand develops.

[2] See p. 1266.

REPORT OF COMMITTEE D-13
ON
TEXTILE MATERIALS

Committee D-13 on Textile Materials has held two meetings during the year, one on October 14 to 16, 1936, in New York City, and the other on March 10 to 12, 1937, at Providence, R. I. The number of members and guests registered at these meetings was 104 and 103, respectively. A third meeting will be held in June at New York City in conjunction with the annual meeting of the Society.

The Advisory Committee has approved the following changes in subcommittee chairmanships: Mr. F. S. Mapes was appointed to fill the vacancy in the chairmanship of Subcommittee A-4 on Asbestos and Its Textile Products, and Mr. A. R. Howe has been made chairman of the reorganized Subcommittee A-5 on Bast Fibers and Their Products. Mr. A. L. Brassell has resigned as chairman of Subcommittee B-2 on Nomenclature and Definitions and Mr. E. D. Fowle has been appointed in his place. The Advisory Committee has discontinued Subcommittee A-7 on Silk with the understanding that it will be retained as inactive until such time as conditions warrant its reorganization. The title of Subcommittee A-5 on Bast Fibers and Their Products has been changed to read "Bast and Leaf Fibers and Their Products." The title of Section III, of Subcommittee B-1, "on Humidity" has been changed to read "on Atmospheric Conditions."

Mr. Ephraim Freedman was appointed to represent the A.S.T.M. at the Trade Practice Conference on the Shrinkage of Woven Cotton Yard Goods held in New York City on March 9, 1937. Mr. Frederick Bonnet and Mr. Ephraim Freedman, as representatives of Committee D-13, served on a committee on arrangements for the National Rayon Technical Conference to be held in Washington, D. C., on May 14 and 15, 1937.

Two of the committee's standard methods, namely, the General Methods of Testing Woven Textile Fabrics (D 39 - 36) and the Method of Test for Shrinkage in Laundering of Woven Cotton Cloth (D 437 - 36), have been approved by the American Standards Association as American standards and assigned the designations L5 - 1936 and L10 - 1936, respectively.

During the year a special committee in charge of Mr. R. H. Brown, acting upon the instructions of the Advisory Committee, prepared a thorough revision of the Regulations Governing Committee D-13. These have been submitted to letter ballot of the membership and accepted.

The committee is planning an exhibit in connection with the annual meeting of the Society in June. Its preparation is in charge of Mr. D. E. Douty. It will be designed to show the nature of the textile materials with which the committee deals and such interesting features of its work as it is possible to display.

The spring meeting was made the occasion of a testimonial dinner to Mr. G. B. Haven. He was presented with a suitably worded scroll, beautifully engrossed in colors. The arrangements were in charge of Mr. D. E. Douty, the first secretary of Committee D-13.

The October meeting was featured by a Symposium on Rayon, and a Symposium on Atmospheric Conditions was held in connection with the March meeting. Of the 25 D-13 subcommittees, 20 held sessions at the fall meeting and 19 at the spring meeting. A new high mark for total attendance at these meetings was established this year. These events may be considered to be one practical measure of the continually growing interest of the members in the work and activities of the committee.

The present membership of the committee is 183; of whom 77 are classified as producers, 51 as consumers, and 55 as general interest members.

RECOMMENDATIONS AFFECTING STANDARDS

The recommendations of the committee regarding the standards and tentative standards under its jurisdiction are summarized below, together with the analysis of the letter ballot on each item. Comments, where necessary, are made under the Activities of Subcommittees.

I. Proposed Tentative Standards.—The committee recommends for publication as tentative the following three new methods of test, appended hereto:

Tentative Methods of Test for Wool Felt,[1]
Tentative Methods of Testing and Tolerances for Certain Wool and Part Wool Fabrics,[2] and
Tentative Method of Test for Volumetric Determination of Small Amounts of Copper in Textiles.[3]

II. Proposed New Standard.—The committee recommends that the Specifications and Methods of Test for Tire Fabrics Other than Cord Fabrics, as appended hereto,[4] be adopted as standard. The proposed standard is a consolidation of the following existing standards:

Standard Methods of Testing and Tolerances for Tire Fabrics Other than Cord Fabrics (D 122 – 33),[5]
Standard Specifications and Methods of Test for Chafer Tire Fabrics (D 316 – 34),[5] and
Standard Specifications and Methods of Test for Enameling Duck for the Tire Industry (D 336 – 33).[5]

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[1] See p. 1226. [2] See p. 1231. [3] See p. 1240.

[4] These revised specifications were adopted as standard by the Society and appear in the 1937 Supplement to Book of A.S.T.M. Standards, p. 244.—ED.

[5] 1936 Book of A.S.T.M. Standards, Part II, pp. 1371, 1309, 1314.

This consolidation involves no modification in substance of these three standards. It is accordingly recommended that the change be effected immediately and the committee accordingly asks for the necessary nine-tenths vote at the annual meeting in order that the consolidated form may be referred to letter ballot of the Society for immediate adoption as standard.

III. Proposed Revisions of Tentative Standards.—The committee is presenting revisions in the following tentative definitions and four methods of test and recommends that, as revised, they be continued as tentative:

Tentative Definitions and Terms Relating to Textile Materials (D 123 - 36 T), revised as appended hereto,[1]
Tentative Methods of Testing and Tolerances for Rayon (D 258 - 35 T), revised as proposed in the Appendix,
Tentative Methods for the Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles (D 276 - 35 T), revised as proposed in the Appendix,
Tentative General Methods of Test for Properties of Cotton Fibers (D 414 - 35 T), revised as proposed in the Appendix, and
Tentative Methods of Testing Pile Floor Covering (D 418 - 36 T), revised as appended hereto.[2]

IV. Proposed Revisions of Standards.—The committee recommends for publication as tentative, revisions of the Standard General Methods of Testing Woven Textile Fabrics (D 39 - 36), as proposed in the Appendix.

The committee is recommending revisions in the following five standards for adoption immediately and accordingly asks for the necessary nine-tenths vote at the annual meeting in order that they may be referred to letter ballot of the Society for immediate adoption as standard:

Standard Specifications for Textile Testing Machines (D 76 - 36), revised as proposed in the Appendix,
Standard General Methods of Testing and Tolerances for Cotton Yarns and Threads (D 180 - 36), revised as proposed in the Appendix,
Standard Specifications and Methods of Test for Asbestos Yarns (D 299 - 36), revised as proposed in the Appendix,
Standard Specifications and Methods of Test for Asbestos Roving for Electrical Purposes (D 375 - 36), revised as appended hereto,[3] and
Standard Specifications and Methods of Test for Asbestos Tape for Electrical Purposes (D 315 - 36), revised as proposed in the Appendix.

V. Adoption of Tentative Standards as Standard.—The committee is proposing revisions in three of the following four tentative standards and recommends that these methods be approved for reference to letter ballot of the Society for adoption as standard:

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[1] See p. 1243. [2] See p. 1235.

[3] These revised specifications and methods of test were adopted as standard by the Society and appear in the 1937 Supplement to Book of A.S.T.M. Standards, p. 255.—ED.

Tentative Methods of Test for Fineness of Wool (D 419 - 35 T),

Tentative Method of Test for Fastness of Dyed or Printed Cotton Fabrics to Laundering or Domestic Washing (D 435 - 36 T), revised as proposed in the Appendix,

Tentative Method of Test for Fastness of Dyed or Printed Silk or Rayon Fabrics to Laundering or Domestic Washing (D 436 - 36 T), revised as proposed in the Appendix, and

Tentative Methods of Test for Small Amounts of Copper and Manganese in Textiles (D 377 - 36 T), revised as appended hereto.[1]

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirm-ative	Neg-ative	Ballots Marked "Not Voting"
I. PROPOSED TENTATIVE STANDARDS			
Methods of Test for Wool Felt	24	0	54
Methods of Testing and Tolerances for Certain Wool and Part Wool Fabrics	28	0	50
Methods of Test for Volumetric Determination of Small Amounts of Copper in Textiles	23	1	54
II. PROPOSED NEW STANDARD			
Specification and Methods of Test for Tire Fabrics Other than Cord Fabrics	26	0	52
III. PROPOSED REVISIONS OF TENTATIVE STANDARDS			
Definitions and Terms Relating to Textile Materials (D 123 - 36 T)	47	5	26
Methods of Testing and Tolerances for Rayon (D 258 - 35 T)	31 31	1 0	46 47
Methods for the Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles (D 276 - 35 T)	46	0	32
General Methods of Test for Properties of Cotton Fibers (D 414 - 35 T)	35	0	43
Methods of Testing Pile Floor Covering (D 418 - 36 T)	20	0	58
IV. PROPOSED REVISIONS OF STANDARDS			
General Methods of Testing Woven Textile Fabrics (D 39 - 36)	59	1	18
Specifications for Textile Testing Machines (D 76 - 36), immediate adoption	58	2	18
General Methods of Testing and Tolerances for Cotton Yarns and Threads (D 180 - 36), immediate adoption	46	0	32
Specifications and Methods of Test for Asbestos Yarns (D 299 - 36), immediate adoption	23	0	55
Specifications and Methods of Test for Asbestos Roving for Electrical Purposes (D 375 - 36), immediate adoption	20	0	58
Specifications and Methods of Test for Asbestos Tape for Electrical Purposes (D 315 - 36), immediate adoption	19	0	59
V. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Methods of Test for Fineness of Wool (D 419 - 35 T)	30	0	48
Method of Test for Fastness of Dyed or Printed Cotton Fabrics to Laundering or Domestic Washing (D 435 - 36 T), as revised	29	0	49
Method of Test for Fastness of Dyed or Printed Silk or Rayon Fabrics to Laundering or Domestic Washing (D 436 - 36 T), as revised	29	0	49
Methods of Test for Small Amounts of Copper and Manganese in Textiles (D 377 - 36 T), as revised	24	0	54
VI. WITHDRAWAL OF STANDARDS			
Methods of Testing and Tolerances for Tire Fabrics Other than Cord Fabrics (D 122 - 33)	31	0	47
Specifications and Methods of Test for Chafer Tire Fabrics (D 316 - 34) with existing tentative revision	28	0	50
Specifications and Methods of Test for Enameling Duck for the Tire Industry (D 336 - 33)	28	0	50

VI. Withdrawal of Standards.—The committee recommends the withdrawal of the following standards:

Standard Methods of Testing and Tolerances for Tire Fabrics Other than Cord Fabrics (D 122 - 33),

Standard Specifications and Methods of Test for Chafer Tire Fabrics (D 316 - 34), and the pending tentative revision, and

Standard Specifications and Methods of Test for Enameling Duck for the Tire Industry (D 336 - 33).

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[1] These revised methods of test were adopted as standard by the Society and appear in the 1937 Supplement to Book of A.S.T.M. Standards, p. 269.—ED.

These three standards have been consolidated in one standard which is being recommended for immediate adoption as standard.

VII. Tentative Standards Continued as Tentative.—The committee recommends that all other tentative standards under its jurisdiction not specifically referred to be continued as tentative for another year.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 185 members; 78 members have returned their ballots, with the results shown in the accompanying Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee A-1 on Cotton and Its Products (K. B. Cook, chairman):

Section I on Cotton (R. W. Webb, chairman).—This section is presenting for publication tentative revisions, as given in the Appendix, of the Tentative General Methods of Test for Properties of Cotton Fibers (D 414 – 35 T) to remove ambiguities, incorporate improvements and to improve the clearness and convenience of use of these methods.

The section is continuing its work of developing more rapid and more practical methods for determining the important fiber properties as well as tests for properties which at present are not included in the methods.

In line with this section's educational program a paper entitled "Application of Certain Chemical Methods to the Determination of Quality of Raw Cotton," by Carl M. Conrad, was presented at the October meeting.

Section II on Cotton Yarns and Threads (A. C. Clifford, chairman).—This section is recommending for immediate adoption as standard a revision, as given in the Appendix, of the Standard General Methods of Testing and Tolerances for Cotton Yarns and Threads (D 180 – 36). This revision changes the tolerances for the standard temperature to the present generally accepted values.

The section is developing a method for the determination of yardage of thread or yarn in a package under uniform and controlled tension by means of a measuring device which it has recently perfected. Work is being continued on an "evenness" test for yarns. A study of the moisture regain of cotton yarns and threads and a survey of testing methods for heavily plied yarns will be undertaken.

Section III on Light and Medium Cotton Woven Fabrics (R. T. Fisher, chairman).—This section is studying the Tentative Methods of Testing and Tolerances for Certain Carded Cotton Gray Goods (D 433 – 36 T) to determine its practicability to users in its present form. Investigations are being conducted on methods for the determination of sizing in gray and finished fabrics.

Section IV on Tire Fabrics (Martin Castricum, chairman).—This section has combined the three existing standard specifications and methods of tests for tire fabrics other than cord fabrics into a single standard, as described earlier in this report, which is recommended for immediate adoption.

The section is undertaking a thorough revision of the remaining standards under its jurisdiction and is continuing its active cooperation with Section II on Machines, of Subcommittee B-1, in the development of a dynamic calibration method for horizontal type testing machines for determining the strength of textiles. An interlaboratory study of the accuracy of present methods of determining twist in single yarns is also under way.

Subcommittee A-2 on Rayon and Its Products (A. M. Tenney, chairman):

This subcommittee sponsored a very successful Symposium on Rayon at the October, 1936, meeting. Several important papers were presented which are listed later in this report under the activities of Subcommittee B-6 on Papers.

Section I on Rayon (A. M. Tenney, chairman).—This section is presenting revisions for publication as tentative, as given in the Appendix, of the Tentative Methods of Testing and Tolerances for Rayon (D 258 - 35 T). These revisions have been made necessary through recent improvements in the technique of testing rayons.

Section II on Rayon Fabrics (Alexis Sommaripa, chairman).—This section is actively engaged in developing methods of testing spun rayon fabrics, particularly with regard to wrinkling and bulging.

Subcommittee A-3 on Wool and Its Products (G. E. Hopkins, chairman):

This subcommittee has authorized certain editorial changes in three of the standards under its jurisdiction, namely, the Standard Method of Estimating Hard Scoured Wool in Wool in the Grease (D 232 - 36),[1] the Standard Methods of Testing and Tolerances for Woolen Yarns (D 403 - 36)[1] and for Worsted Yarns (D 404 - 36).[1] The terms "free fat" and "soap fat" are to be changed to "ether extract" and "alcohol extract," respectively. These changes are intended to designate more accurately the fat components present in the materials involved, and have been approved by letter ballot of the subcommittee.

Section I on Wool (Werner Von Bergen, chairman).—This section is recommending the adoption as standard of the Tentative Methods of Test for Fineness of Wool (D 419 - 35 T).

The section is working on the development of methods for determination of shrinkage in grease wool, length of wool top, and identification and grading of mohair and other specialty hair fibers.

Section II on Felt (J. F. Marshall, chairman).—This section is presenting proposed Tentative Methods of Test for Wool Felt, as appended hereto,[2] These methods were developed to meet a demand from the industry.

Section III on Woolen and Worsted Yarns (Fred Noechel, chairman).—This section is reviewing the present standards under its jurisdiction and developing methods of testing and tolerances for yarn spun from wool mixed with other fibers. A study is being made of the number of tests necessary to properly evaluate the breaking strength of a yarn.

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[1] 1936 Book of A.S.T.M. Standards, Part II, pp. 1385, 1388, 1393.

[2] See p. 1226.

Section IV on Pile Floor Coverings (A. G. Ashcroft, chairman).—This section is presenting for publication revisions, as appended hereto,[1] of the Tentative Methods of Testing Pile Floor Covering (D 418 – 36 T) to include provisions for color fastness to light. The section is working on the development of wear test methods, chemical tests, methods for tuft bind measurement, definitions, and revision of standards to include statistical data on precision of test methods.

Section V on Woolen and Worsted Fabrics (F. A. Prisley, chairman).—This section is presenting proposed Tentative Methods of Testing and Tolerances for Certain Wool and Part Wool Fabrics which, in part, agree in substance with the Recommended Commercial Standard for Wool and Part Wool Fabrics (TS – 2300) promulgated by the U. S. Department of Commerce. These methods fill a need of the wool and worsted industry for methods supplementary to the present Standard General Methods of Testing Woven Textile Fabrics (D 39 – 36). The section has further test methods under consideration.[2]

Subcommittee A-4 on Asbestos and Its Textile Products (F. S. Mapes, chairman):

This subcommittee is recommending for immediate adoption revisions of the Standard Specifications and Methods of Test for Asbestos Yarns (D 299 – 36), for Asbestos Roving for Electrical Purposes (D 375 – 36), and for Asbestos Tape for Electrical Purposes (D 315 – 36). The changes reduce the chemical test specimen for the determination of asbestos content to a more workable size.

The subcommittee is undertaking the development of a resistivity test for asbestos materials for electrical purposes, a revision of the Standard Specifications D 315 to include data for 10-mil tape, and a survey of test methods for asbestos brake linings and woven asbestos cloth.

Subcommittee A-5 on Bast Fibers and Their Products (A. R. Howe, chairman):

This subcommittee is actively at work gathering fundamental data preparatory to developing methods of test and definitions. Studies are being carried out on breaking strengths, fiber length determination, and numbering of jute yarns.

Subcommittee A-6 on Household and Garment Fabrics (Ruth O'Brien, chairman):

This subcommittee is working toward the development of a standard system of classification and nomenclature of fabrics, the establishment of standard definitions for household fabrics, and a standard system for rating qualities of woven dress fabrics.

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[1] See p. 1235.

[2] See Editorial Note, p. 490.—ED.

Subcommittee B-1 on Methods and Machines (W. D. Appel, chairman):

Section I on Methods (W. D. Appel, chairman).—This section is recommending revisions for publication as tentative, as given in the Appendix, of the Standard General Methods of Testing Woven Textile Fabrics (D 39 - 36) to provide needed methods for determination of bow in fabrics and twist in yarns removed from fabrics. The addition of the sulfuric acid method of determining wool in mixtures of wool and cotton, appended hereto,[1] to the Tentative Methods for the Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles (D 276 - 35 T) is being recommended.

The section is studying methods for tearing strength of fabrics and for the quantitative analysis of fiber mixtures.

This section, together with Section II, is sponsoring a Symposium on Testing Methods for the October, 1937, meeting of Committee D-13.

Section II on Machines (H. J. Ball, chairman).—This section is recommending, for immediate adoption, a revision of the Standard Specifications for Textile Testing Machines (D 76 - 36), as proposed in the Appendix, which consists of a clarification of the specification for single-strand clamps and the addition of a performance requirement for the determination of flatness and parallelism of the jaw surfaces.

The section is studying the effect of rate of application of load, when using Type *B* machines, on strength and elongation of cotton yarns. The work on specifications for diaphragm bursting testers is being continued. Specifications for the other textile testing machines, such as twist and crimp testers, yarn reels, numbering scales, drying ovens, etc., will be developed.

Section III on Atmospheric Conditions (R. H. Brown, chairman).—This section is reviewing the existing standards and tentative standards with respect to requirements for atmospheric conditions and regains. A study is being made of the time for test specimens to reach equilibrium in the standard atmosphere.

The section sponsored a very successful Symposium on Atmospheric Conditions at the March, 1937, meeting. The titles of the symposium papers are listed under the report of Subcommittee B-6 on Papers.

Subcommittee B-2 on Nomenclature and Definitions (A. L. Brassell, chairman):

This subcommittee recommends a revision, as appended hereto,[2] of the Tentative Definitions and Terms Relating to Textile Materials (D 123 - 36 T). By this revision several new definitions are added, relating chiefly to wool. The subcommittee also has a number of additional definitions under consideration.

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[1] See p. 1187.

[2] The new and modified terms have been incorporated in the revised tentative definitions which appear on p. 1243.—ED.

Subcommittee B-4 on Bleaching, Dyeing and Finishing (W. M. Scott, chairman):

This subcommittee is presenting for publication as tentative the proposed Tentative Method of Test for Volumetric Determination of Small Amounts of Copper in Textiles, as appended hereto.[1] This method is a revision of the procedure now appearing in the Tentative Methods of Test for Small Amounts of Copper and Manganese in Textiles (D 377 – 36 T). Revisions of the colorimetric tests for copper and manganese in textiles in the Tentative Methods D 377 are also proposed, and these changes have been incorporated in the Methods for Colorimetric Determination of Small Amounts of Copper and Manganese in Textiles, appended hereto,[2] which are recommended for adoption as standard.

The subcommittee is also presenting revisions, as given in the Appendix, of the Tentative Method of Test for Fastness of Dyed or Printed Cotton Fabrics to Laundering or Domestic Washing (D 435 – 36 T) and for Fastness of Dyed or Printed Silk or Rayon Fabrics to Laundering or Domestic Washing (D 436 – 36 T) and is recommending their adoption as standard.

The subcommittee is developing methods of test for water resistance and fastness to light, and a general method for determining the acidity or alkalinity of textiles.

The subcommittee will sponsor a Symposium on Finishing to be held at the March, 1938, meeting of Committee D-13.

Subcommittee B-5 on Sampling, Presentation and Interpretation of Data (A. G. Ashcroft, chairman):

This subcommittee is working on procedures by which the precision of test methods and number of test specimens required may be specified. Illustrative papers and articles will be presented periodically.

Two reports were given at the March, 1937, meeting as examples of methods of determining precision of a test method. These were entitled "Statistical Interlaboratory Check on Tuft Length Measurement of Pile Floor Coverings," by J. N. Dow, and "The Precision of the Method for Measuring Back Thickness of Pile Floor Coverings," by O. P. Beckwith.

Subcommittee B-6 on Papers (Dean Harvey, chairman):

This subcommittee arranged for the presentation of the following papers at the interim meetings of Committee D-13:

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[1] See p. 1240.

[2] These methods were adopted as standard by the Society and appear in the 1937 Supplement to Book of A.S.T.M. Standards, p. 269.—ED.

"The Testing of Rayon Yarns," by A. Stuart Hunter,
"Rayons as Consumers View Them," by Ephraim Freedman,
"Stereo-Radiography of Textiles," by H. F. Sherwood,
"The Use of Statistical Methods as an Aid to the Control of Quality of Materials," by Dean Harvey,
"Importance of Atmospheric Conditions in Testing Textile Materials," by R. H. Adams,
"Importance of Atmospheric Conditions in Testing Wool," by A. G. Ashcroft,
"Effect of Atmospheric Conditions in Testing Rayon," by A. G. Scroggie,
"The Role of Atmospheric Conditions in the Testing of Asbestos Textiles," by David Wolochow,
"The Mechanics of Moisture Absorption by Textiles," by A. C. Walker, and
"Regain in Textile Materials," by R. H. Brown.

Arrangements were also made for formal discussions of Dean Harvey's paper by G. E. Hopkins, O. P. Beckwith, A. G. Scroggie and L. R. Jones who presented, by means of actual data, illustrations of the use of statistical methods in quality control; and for discussion of R. H. Brown's paper by G. E. Hopkins, W. D. Appel and A. C. Clifford. Abstracts of the above papers are being prepared for publication in the 1937 edition of the volume of "A.S.T.M. Standards for Textile Materials." The subcommittee is planning a Symposium on Testing Methods for October, 1937, and a Symposium on Finishing for March, 1938.

This report has been submitted to letter ballot of the committee which consists of 185 members; 78 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. J. BALL,
Chairman.

W. H. WHITCOMB,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting Committee D-13 on Textile Materials presented to the Society through Committee E-10 on Standards proposed Tentative Specifications and Methods of Test for Fineness of Wool Tops. These specifications were accepted by Committee E-10 on August 26, 1937, and appear on p. 1218.

APPENDIX

PROPOSED REVISIONS IN STANDARDS FOR TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentative standards covering textile materials which are referred to earlier in this report. In connection with each title is given the reference to the publication in which the standard appears in its present form.

Proposed Revisions of Tentative Standards

Revision of Tentative Methods of Testing and Tolerances for Rayon (A.S.T.M. Designation: D 258 – 35 T):[1]

Section 2 (a).—Change this section to read as follows by the omission of the words in brackets:

2. (*a*) *Rayon.*—A generic term for filaments made from various solutions of modified cellulose by pressing [or drawing] the cellulose solution through an orifice, and solidifying it in the form of a filament.

Section 5.—Change this section to read as follows by the addition of the italicized words:

5. The denier of a rayon yarn is ***numerically equal to*** the weight in grams of 9000 m.

Section 10 (b).—Change this section from its present form: namely,

(*b*) Ultimate tensile strength, or tensile strength at the highest yield point, may be specified.

to read as follows:

(*b*) Tensile strength at break, or at the yield point, may be specified. Tensile strength shall be expressed as the number of grams per denier of the yarn before testing.

Section 13 (c).—Add the following sentence at the end of the Note following this section:

The methods given in this section for removing "other foreign substances" are not suitable for complete removal of materials insoluble in these solvents.

Section 14 (a).—Change this section and the Note to read as follows by transferring the third and fourth sentences of this section to the end of the Note and omitting the words in brackets, relettering Paragraph (*b*) as (*a*):

(*b*) If sufficient sample of rayon yarn is available, the denier and the filament count shall be determined. These values shall be compared with the various numbers offered for sale commercially by the different manufacturers of rayon.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1233 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 1213.

NOTE.—This test is valuable only when the sample is known to be material of fairly recent manufacture and is known to be a commercial product, not an experimental lot. Occasionally, [as in the case of very fine denier] this will definitely determine the type of rayon yarn. At other times, it merely limits the number of possibilities and, on occasion, is of no assistance.

Section 15 (*a*).—Change the last sentence of the last paragraph in this section to read as follows by the addition of the italicized words and the omission of those in brackets:

Partially saponified or hydrolyzed cellulose-acetate rayon [partly dissolves] *may be partly or completely insoluble* in the solvents described above.

Section 17 (*b*).—At the end of the first sentence in this section insert the phrase "suitable sectioning device" in place of the words "Schwarz sectioning plate"; also omit the footnote.

Change the second paragraph of this section to read as follows by the addition of the italicized words and the omission of the word in brackets:

Viscose rayon *usually* has an irregular serrated edge, cuprammonium rayon is almost round and has a [serrated] *smooth, unserrated* edge.

Section 19.—Change the first sentence of this section to read as follows by the addition of the italicized words and the omission of those in brackets:

A test skein of 200 turns of yarn shall be reeled, using any reel employing a straight wind traverse having a perimeter of 112.5 cm. [may be used]

Change the second sentence of the Note under this section to read as follows by the addition of the italicized words and the omission of the word in brackets:

Each skein [must] *may* contain 200 turns as before *if strength tests are to be subsequently made on it.*

Add the following paragraph at the end of the Note:

When test skeins of 200 turns of yarn are not required for the strength test, the length of skeins reeled on the cotton reel may be 246 yd. (225 m.), or 492 yd. (450 m.) if desired. These skeins shall be weighed in accordance with Section 19.

Section 25.—Add the following sentence at the end of this section:

Denier skeins to be used for the strength test shall be removed from the reel *after* collapsing the reel sufficiently to prevent stretching or damage before being placed in the tensile testing machine.

Section 26.—Change this section from its present form: namely,

26. The test skeins shall be conditioned for at least 3 hr. in an atmosphere, kept in rapid motion by an electric fan, having a relative humidity of 65 per cent at 70 F. (21 C.).

to read as follows:

26. (*a*) The test skeins shall be conditioned to a moisture equilibrium in an atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of ±2 per cent is permitted in relative humidity and plus 10 F. (6 C.) in temperature.

(*b*) It shall be considered that moisture equilibrium is reached when, after free exposure to the atmosphere kept in rapid motion by an electric fan, there is no progressive increase in weight. Moisture equilibrium shall be approached from the dry side (not moisture free).

NOTE.—It is believed that test skeins will reach moisture equilibrium after such exposure for a 3-hr. period.

Section 28.—Change the first sentence of this section to read as follows by the addition of the italicized word and the omission of the phrase in brackets:

[Immediately after conditioning (Section 26),] The *conditioned* test skeins shall be separately broken on the testing machine.

Sections 30, 31 and 32.—Change these sections from their present form to read as follows:

30. *Apparatus.*—(*a*) A single strand tester of proper capacity with the jaws set 18 in. between grips shall be used.

(*b*) The speed of the pulling jaw of a pendulum type testing machine shall be 12 ±½ in. per min.

(*c*) The speed of operation of a constant specimen-rate-of-load type testing machine shall be such that the rate of application of the load shall be 4 g. per denier per min.

31. *Procedure, Alternate Method.*—Five single strands from each of 10 spools, cops, tubes, cones, or skeins shall be broken after conditioning the package in accordance with Section 26.

NOTE.—It is believed that the packages will reach moisture equilibrium after conditioning in accordance with Section 26 for a 3-hr. period for skeins and for a 12-hr. period for any other form of package.

32. *Number of Tension Tests.*—The average of 50 tests free from breaks at the jaws shall be the strength.

Revision of Tentative Methods for the Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles (D 276 – 35 T):[1]

New Sections.—Add as new Sections 9 and 10 the following method for the determination of wool and cotton in wool-cotton mixtures:

Wool, Sulfuric Acid Method[2]

9. (*a*) The residue from the test for regenerated cellulose rayon (Section 6), which contains cotton and wool, shall be immersed in 200 ml. of a boiling sulfuric acid solution (which has been carefully prepared to contain 1 per cent of H_2SO_4 by weight) for 7 to 10 min. It shall then be transferred to a crucible having a medium fritted glass bottom and the excess acid solution shall be removed by suction. (A Gooch crucible, without asbestos, may be used to advantage for the filtrations if the liquors passing through at first are returned to the crucible so that all fibers are collected in the pad which forms in the crucible.) The specimen shall be immersed in 200 ml. of a solution of sulfuric acid (which has been carefully prepared to contain 70 per cent of H_2SO_4 by weight) at a temperature of 43 C. for 10 min. The undissolved fibers shall be collected in the crucible and thoroughly washed with cold water, immersed in a solution of sodium bicarbonate (2 per cent) at room temperature for 5 min. to neutralize any acid, collected again in the crucible, washed, and dried at 105

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1215 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 1172.

[2] This method conforms in substance with the method developed by the American Association of Textile Chemists and Colorists.

to 110 C. to constant weight. The weight of the dried residue from this treatment is weight H.

(*b*) If it is not necessary to subject the specimen to the procedures given in Sections 3 to 6, inclusive, the specimen shall be prepared and treated in accordance with Paragraph (*a*) with the following exceptions: Tightly woven, knit, or felt material shall be cut into strips $\frac{1}{8}$ to $\frac{1}{4}$ in. in width. Highly twisted yarns shall be partially untwisted. A specimen weighing approximately 2 g. shall be taken for the test. It shall be dried to constant weight at 105 to 110 C. in accordance with Section 3 (*b*). The weight of the dried specimen is weight B.

(*c*) The wool content of the specimen shall be calculated from the following formula:

$$\text{Wool (dry basis), per cent} = \frac{H}{B} \times 100$$

where B = the weight of the dry specimen (Section 3 (*b*)), and
H = the weight of the dry specimen after removal of cotton.

Cotton

10. The cotton content of the specimen shall be calculated from either Eq. (1), if the specimen has been treated in accordance with Sections 3 to 6 inclusive, or Eq. (2), if the specimen has been analyzed only for wool and cotton, as follows:

$$\text{Cotton (dry basis), per cent} = \frac{F - H}{B} \times 100 \quad \text{(1)}$$

$$\text{Cotton (dry basis), per cent} = \frac{B - H}{B} \times 100 \quad \text{(2)}$$

where B = the weight of the dry specimen (Section 3 (*b*)),
F = the weight of the dry residue (Section 6 (*a*)), and
H = the weight of the dry specimen after removal of cotton.

Revision of Tentative General Methods of Test for Properties of Cotton Fibers (A.S.T.M. Designation: D 414 – 35 T):[1]

Section 16 (b).—Change this section to read as follows by the addition of the italicized words:

(*b*) The mount shall be placed on the mechanical stage of the microscope ***which shall be equipped with a filar micrometer eyepiece.***

Section 17.—Change this section to read as follows by the addition of the italicized figure and the omission of that in brackets:

17. The fibers shall be examined near their mid-portion at a magnification of approximately [100] *400* diameters.

Section 18 (b).—Change this section from its present form: namely,

(*b*) For the purpose of this test, thin-walled or immature fibers are those that upon treatment with 18 per cent caustic soda exhibit many spirals. (Figure 5 shows photomicrographs of thin-walled or immature fibers as compared with medium and thick-walled fibers. The appearance of these fibers before and after treatment is shown for purposes of reference.)

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1202 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 1186.

to read as follows:

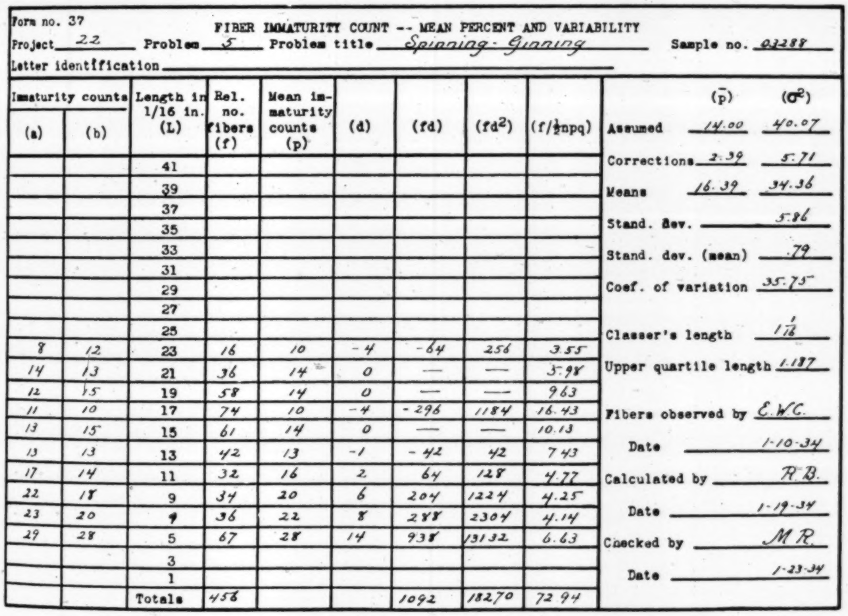
(*b*) For the purpose of this test, thin-walled or immature fibers shall be those that upon treatment with NaOH solution (18 per cent) show a fiber wall less than one-half the width of the adjacent lumen at the widest part of the fiber ribbon, that is, midway between two edge-on positions of the ribbon. All other fibers shall be classed as mature or thick-walled fibers. Doubtful classification shall be decided with the aid of the filar micrometer. (Figure 5 shows photomicrographs of thin-walled or immature fibers as compared with thick-walled fibers. The appearance of these fibers before and after treatment is shown for purposes of reference.)

Figures 1 and 2.—Delete the following from the record forms shown in these figures:

Variability of the Middle 80 Percentile . . . .%
Variability of the Middle 50 Percentile . . . .%

Figure 3.—Change the word "coefficient of variability" which appears in the record form in this figure to read "coefficient of variation."

Figure 4.—Replace this figure, which shows a facsimile of record form with data and calculations for determination of the immaturity count, by the accompanying Fig. 1.



Form no. 37
FIBER IMMATURITY COUNT -- MEAN PERCENT AND VARIABILITY
Project 22 Problem 5 Problem title Spinning - Ginning Sample no. 03288
Letter identification

Immaturity counts (a)	Immaturity counts (b)	Length in 1/16 in. (L)	Rel. no. fibers (f)	Mean immaturity counts (p)	(d)	(fd)	(fd^2)	$(f/\frac{1}{2}npq)$
		41						
		39						
		37						
		35						
		33						
		31						
		29						
		27						
		25						
8	12	23	16	10	-4	-64	256	3.55
14	13	21	36	14	0	—	—	5.98
12	15	19	58	14	0	—	—	963
11	10	17	74	10	-4	-296	1184	16.43
13	15	15	61	14	0	—	—	10.13
13	13	13	42	13	-1	-42	42	7 43
17	14	11	32	16	2	64	128	4.77
22	18	9	34	20	6	204	1224	4.25
23	20	9	36	22	8	288	2304	4.14
29	28	5	67	28	14	938	13132	6.63
		3						
		1						
		Totals	456			1092	18270	72 94

	$(\bar{p})$	(σ^2)
Assumed	14.00	40.07
Corrections	2.39	5.71
Means	16.39	34.36

Stand. dev. 5.86
Stand. dev. (mean) .79
Coef. of variation 35.75

Classer's length $1\frac{1}{16}$
Upper quartile length 1.187

Fibers observed by E.W.C.
Date 1-10-34
Calculated by R.B.
Date 1-19-34
Checked by M.R.
Date 1-23-34

FIG. 1.—Facsimile of Record Form with Data and Calculations for Determination of the Immaturity Count.

Table I.—Change this table on corrections applicable to machine breaks for cottons from its present form to read as shown in the accompanying Table I.

TABLE I.—CORRECTIONS APPLICABLE TO MACHINE BREAKS FOR COTTONS HAVING DIFFERENT MEAN CORRECTED MACHINE BREAKS[a] AND CIRCUMFERENCES DEVIATING FROM STANDARD CIRCUMFERENCE.[b] [c]

Deviation of Circumference from Standard, in.	Corrections in Pounds Applicable to Cottons Having Average Corrected Machine Break of														
	60 lb.	65 lb.	70 lb.	75 lb.	80 lb.	85 lb.	90 lb.	95 lb.	100 lb.	105 lb.	110 lb.	115 lb.	120 lb.	125 lb.	130 lb.
0.001.......	0	1	1	1	1	1	1	1	1	1	1	1	1	2	2
0.002.......	1	1	1	1	2	2	2	2	2	2	3	3	3	3	3
0.003.......	1	2	2	2	2	3	3	3	3	4	4	4	4	5	5
0.004.......	2	2	3	3	3	4	4	4	5	5	5	6	6	6	7
0.005.......	2	3	3	4	4	4	5	5	6	6	7	7	7	8	8
0.006.......	3	3	4	4	5	5	6	6	7	7	8	8	9	9	10
0.007.......	3	4	4	5	6	6	7	7	8	9	9	10	10	11	12
0.008.......	4	4	5	6	6	7	8	8	9	10	10	11	12	12	13
0.009.......	4	5	6	6	7	8	9	9	10	11	12	13	13	14	15
0.010.......	5	5	6	7	8	9	10	11	11	12	13	14	15	16	16

[a] The mean corrected machine break is obtained by a first approximation, by allowing 1 lb. for each 0.001-in. deviation of the mean circumference from standard circumference (0.125 in.). This determines the proper column for the corrections.

[b] Bundles smaller than standard circumference require that the corrections be added; those larger than standard require that the corrections be subtracted.

[c] The corrections are calculated by means of the formula $R = 0.01690\ B_s - 0.55$; where B_s is the mean machine break for bundles of standard circumference, and R is the correction for each 0.001-in. deviation of circumference from standard.

Revision of Tentative Method of Test for Fastness of Dyed or Printed Cotton Fabrics to Laundering or Domestic Washing (A.S.T.M. Designation: D 435-36 T):[1]

Section 4.—Change this section to read as follows by the addition of the italicized words and the omission of those in brackets:

4. The soap solution shall contain 5 g. of 88 per cent soap and 2 g. of 58 per cent soda ash per liter. The soap shall be a good grade of neutral [white chip] *soda soap*, as free as possible from all substances other than true soap. [Although] The proportion of soap is calculated on the basis of *an* 88 per cent soap *chip*, *but* an equivalent amount of a soap having a higher or lower strength may be used. Distilled water, or water of approximately zero hardness (*not over 5 p.p.m.*), shall be used to dissolve the soap.

Section 5.—Change the last sentence of Paragraph (*a*) of this section to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

The specimens shall then be removed from the jars, rinsed [thoroughly in warm distilled water 100 to 110 F. (38 to 43 C.)] *carefully* to insure complete removal of the soap, *in three changes of water of approximately zero hardness at 100 F. ±2 F. (38 C. ± 1 C.), allowing not over 1 min. for each rinsing and squeezing between rinsings*, and *then* wrapped in a clean white towel or similar cloth.

Add the following sentence at the end of Paragraph (*b*) of this section:

The operations of rinsing and drying shall be performed with as little time interval between each operation as possible.

Section 7 (b).—Change the Note following this section to read as follows by addition of the italicized words and the omission of those in brackets:

NOTE.—Standard dyeings [have been prepared by] *are obtainable from the Secretary of* the American Association of Textile Chemists and Colorists [and are available for general use].

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1140 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 1199.

Revision of Tentative Method of Test for Fastness of Dyed or Printed Silk or Rayon Fabrics to Laundering or Domestic Washing (A.S.T.M. Designation: D 436 - 36 T):[1]

Title.—Change the title to read as follows by the addition of the italicized word:

Tentative Method of Test for Fastness of Dyed or Printed *Wool*, Silk or Rayon Fabrics to Laundering or Domestic Washing.

Section 1.—Add the word "wool" before silk.

Section 2 (b).—Change the last sentence of this section to read as follows by the addition of the italicized words and the omission of those in brackets:

To the test specimen shall be sewed pieces, [1-in. square] *approximately 1 sq. in. in size*, of unsized bleached cotton and undyed silk, wool, rayon and cellulose acetate fabrics.

Section 4.—Change this section to read as follows by the addition of the italicized words and the omission of those in brackets:

4. The soap solution shall contain 5 g. of 88 per cent soap per liter. The soap shall be a good grade of neutral [white chip] *soda soap*, as free as possible from all substances other than true soap. [Although] The proportion of soap is calculated on the basis of *an* 88 per cent soap *chip*, *but* an equivalent amount of a soap having a higher or lower strength may be used. Distilled water, or water of approximately zero hardness (*not over 5 p.p.m.*), shall be used to dissolve the soap.

Section 5.—Make the same change in Paragraph (*a*) of this section as recommended above in Section 5 (*a*) of Tentative Method D 435 - 36 T.

Change Paragraph (*b*) of this section to read as follows by the addition of the italicized words and the omission of those in brackets:

(*b*) Drying.—The excess water shall be removed by centrifuging or squeezing. The specimens shall be taken out of the towel and *if composed of silk or rayon they shall be* dried by pressing with a moderately warm, not hot, iron or a flat-plate ironer set to about 275 F. (135 C.). *If the specimens are composed of wool they shall be dried in a current of air at room temperature.* After [ironing] *drying*, the specimens shall be allowed to condition in the open air for 2 hr., or until the colors reach a stable condition. *The operations of rinsing and drying shall be performed with as little time interval between each operation as possible.*

Section 7 (b).—Make the same change in the Note following this section as recommended above in the Note following Section 7 (*b*) of Tentative Specifications D 435 - 36 T.

Proposed Revisions of Standards, Immediate Adoption

Revision of Standard Specifications for Textile Testing Machines (A.S.T.M. Designation: D 76 - 36):[2]

Section 4.—Change this section from its present form: namely,

4. The jaws or clamps for testing a specimen of fabric shall have smooth, flat, metallic, gripping surfaces, with the edges rounded to a radius of not over $\frac{1}{64}$ in. to

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1154 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 1232.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 1291.

prevent cutting. One gripping surface shall be hinged or swiveled, and the other shall be rigidly connected to the frame of the jaw. The pressure between the gripping surfaces shall be secured by any suitable mechanical device so constructed as to grip the specimen firmly before the testing load is applied and to prevent slippage during the progress of the test. The dimension of all gripping surfaces parallel to the direction of application of the load shall be 1 in.; the dimension perpendicular to this direction shall be as specified for the particular type of test under consideration.

to read as follows:

4. The clamps for holding a specimen of fabric shall have metallic gripping surfaces sufficiently smooth, flat and parallel so as to prevent the test specimen from slipping or moving between the gripping surfaces when held under the pressure normal to operation. The dimension of all gripping surfaces parallel to the direction of application of the load shall be 1 in.; the dimension perpendicular to this direction shall be as specified for the particular type of test under consideration. All edges which might cause a cutting action shall be rounded to a radius of not over $\frac{1}{64}$ in. The design of the clamp shall be such that one gripping surface shall be an integral part of the rigid frame of the clamp, while the other shall be on a part hinged or swiveled to the movable member of the clamp. The pressure between the gripping surfaces, sufficient to clamp the specimen firmly before the testing load is applied and to prevent slippage during the progress of the test, shall be secured by any suitably constructed mechanical device operating on the movable member of the clamp.

Note.—As a practical method of determining the degree of flatness and parallelism of the assembled clamp mechanism, it is recommended that a sheet of thin white paper, between two thin sheets of carbon paper, be placed between the gripping surfaces, and the jaws then brought together with a light pressure.

Revision of Standard General Methods of Testing and Tolerances for Cotton Yarns and Threads (A.S.T.M. Designation: D 180 – 36):[1]

Section 2 (*b*).—Change the last sentence of this section to read as follows by the addition of the italicized figures and the omission of the word and figures in brackets:

A tolerance of ±2 per cent is permitted in relative humidity and [plus 10 F. (6 C.)] *±2 F.* (*1.1 C.*) in temperature.

Revision of Standard Specifications and Methods of Test for Asbestos Yarns (A.S.T.M. Designation: D 299 – 36):[2]

Section 10.—Change the first sentence of Paragraph (*a*) to read as follows by the addition of the italicized word and figure and the omission of the word and figure in brackets:

Five test specimens, each weighing not less than [200 grains] *5 g.*, shall be dried to constant weight in an oven at 100 to 105 C. (212 to 220 F.), and the weights of the dried specimens recorded.

Revision of Standard Specifications and Methods of Test for Asbestos Tape for Electrical Purposes (A.S.T.M. Designation: D 315 – 36):[3]

Section 15.—Make the same change in the first sentence of Paragraph (*a*) of this section as is recommended above in Specifications D 299 – 36.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1351.

[2] Ibid., p. 1320.

[3] *Ibid.*, p. 1324.

Proposed Tentative Revision of Standard

Tentative Revision[1] of Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39 – 36):[1]

Section 11.—Change this section from its present form: namely,

11. *Raveled Strip Method.*—This test shall be made in accordance with the directions for the grab method (Section 10) with the following exceptions: The specimens shall be 1¼ in. in width if there are 50 or more yarns per inch, and 1½ in. in width if there are less than 50 yarns per inch. Each specimen shall be raveled to 1 in. in width by taking from each side approximately the same number of yarns. The clamps used on the testing machine shall have faces measuring 1 by 1½ in. or more, the longer dimension being perpendicular to the direction of application of the load.

to read as follows:

11. *Raveled Strip Method.*—This test shall be made in accordance with the directions for the grab method (Section 10) with the following exceptions: This method is applicable only to fabrics containing 50 or more yarns per inch. Each specimen shall be 1¼ in. in width and shall be raveled to 1 in. in width by taking from each side approximately the same number of yarns. The clamps used on the testing machine shall have faces measuring 1 by 1½ in. or more, the longer dimension being perpendicular to the direction of application of the load.

New Sections.—Add the following paragraphs at the end of these methods as new Sections 16 and 17:

16. *Twist, Warp and Filling Yarns:* (*a*) *Test Specimens.*—Specimens of the fabric shall measure at least 30 in. in the warp direction for the determination of the warp twist, and at least 30 in. in the filling direction, or the full width of the fabric, for the determination of the filling twist. In all operations care must be exercised to avoid any change in the twist of the yarn to be tested. The yarns may be removed from the fabric in the following manner:

Holding the yarn to be tested at one end, remove it from the fabric for a distance of approximately 10 in. (cutting the cross yarn, if necessary) and grip it at this point in one clamp of the twist tester. Remove the yarn for an additional 10 in., straighten it by applying just sufficient tension to remove the crimp (Section 8) and then grip it in the other clamp of the twist tester. The distance between clamps shall be 10 in.

(*b*) *Procedure.*—The twist of the specimen prepared as directed in Paragraph (*a*) shall be determined by one of the following procedures of the American Society for Testing Materials:

Cotton Yarn.—Section 12 of the Standard General Methods of Testing and Tolerances for Cotton Yarns and Threads (A.S.T.M. Designation: D 180).[2]

Woolen Yarn.—Section 12 of the Standard Methods of Testing and Tolerances for Woolen Yarns (A.S.T.M. Designation: D 403).[3]

Worsted Yarn.—Section 12 of the Standard Methods of Testing and Tolerances for Worsted Yarns (A.S.T.M. Designation: D 404).[4]

(*c*) *Number of Tests.*—One twist test on each of ten or more different warp yarns shall be made and the average of these ten or more tests shall be the average twist of the warp yarns. One twist test on each of ten or more different filling yarns, taken from locations in the fabric as far apart as practicable, shall be made and the average of these ten or more tests shall be the average twist of the filling yarn.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1334.

[2] *Ibid.*, p. 1351.

[3] *Ibid.*, p. 1388.

[4] *Ibid.*, p. 1393.

17. *Bow.*—The fabric shall be laid out smooth on a horizontal surface without tension. The position of a filling yarn in the fabric shall be marked with a line drawn on the fabric across its full width, or by other suitable means. (Marking is not necessary when distinctive colored picks are woven into the fabric at regular intervals.) A straight edge shall be placed across the fabric between the ends of this line at the edges of the fabric. The greatest distance between the line and the straight edge, measured parallel to the edges of the fabric, and the width of the fabric along the straight edge shall be measured. Similar measurements shall be made in at least three different places along a roll or piece, preferably at points 10 yd. from each end and in the center. The bow shall be expressed as a percentage of the width of the fabric and shall be the average for the three or more sets of measurements as calculated from the following formula:

$$\text{Bow, per cent} = \frac{d}{w} \times 100$$

where d = the greatest distance in inches between the marked filling yarn and the straight edge, and

w = the width of the fabric in inches.

REPORT OF COMMITTEE D-17
ON
NAVAL STORES

Committee D-17 on Naval Stores has held no meeting during the year and the work of the committee has accordingly been conducted entirely by correspondence.

Advice has been received from Committee D-6 on Paper and Paper Products (which is now in course of organization) that that committee does not anticipate work on size making value of rosin but the thought has been expressed that Committees D-6 and D-17 should cooperate on matters relating to the use of rosin in size making.

Work is in progress in the laboratory of the chairman on two matters suggested at the last meeting of the committee, that is, on the determination of ash in rosins, and determination of the extent of darkening of rosin on heating under the conditions and for the time rosin would be heated in making varnish. No formal report can be made at this time on either subject, but it is hoped to present some data at the meeting of the committee to be held during the annual meeting of the Society.

Subcommittee IV on Acid and Saponification Number and Unsaponifiable Matter (C. C. Zeigler, chairman) has been very active during the year and is recommending that a proposed Tentative Method of Test for Acid Number of Rosin, and a Method of Test for Saponification Number of Rosin be accepted for publication as tentative, as appended hereto.[1] A report of this subcommittee describing the methods employed and giving the results of its work is appended to this report.

Some additional work has been done on the matter of viscosity of rosin, but no report can be made at this time.

RECOMMENDATIONS AFFECTING STANDARDS

Proposed Tentative Standards.—The committee recommends that the following two methods be accepted for publication as tentative as appended hereto,[1] as proposed by Subcommittee IV on Acid and Saponification Numbers and Unsaponifiable Matter:

Tentative Method of Test for Acid Number of Rosin, and
Tentative Method of Test for Saponification Number of Rosin.

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[1] See pp. 835 and 836.

This recommendation has been submitted to letter ballot of the committee which consists of 17 members; 17 members returned their ballots, the results being as follows:

Items	Affirmative	Negative	Ballots Marked "Not Voting"
PROPOSED TENTATIVE STANDARDS			
Method of Test for Acid Number of Rosin	17	0	0
Method of Test for Saponification Number of Rosin	17	0	0

This report has been submitted to letter ballot of the committee which consists of 17 members; 17 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. P. VEITCH,
Chairman.

C. F. SPEH,
Secretary.

REPORT OF SUBCOMMITTEE IV ON ACID AND SAPONIFICATION NUMBER AND UNSAPONIFIABLE MATTER

Subcommittee IV has been engaged during the past year in a further study of methods for the determination of acid number and saponification number of rosin. Suggestions developed on the basis of the subcommittee's previous experience[1] resulted in the drafting of a new set of methods for these determinations which were sent to subcommittee members, together with samples of WW, N and H rosin. These samples were examined in the laboratories of eight members. Two modifications were used in the case of the acid number determination, a hot and a cold titration. The hot titration consisted of dissolving the rosin by heating the neutral alcohol solvent to the boiling point and then titrating the hot solution. In the cold titration, the rosin was dissolved in the alcohol at room temperature and the titration made at that temperature.

Table I.—Results of Cooperative Tests for Acid Number and Saponification Number of Rosin.

Laboratory	Acid Number (Cold Titration)			Acid Number (Hot Titration)			Saponification Number		
	WW Rosin	N Rosin	H Rosin	WW Rosin	N Rosin	H Rosin	WW Rosin	N Rosin	H Rosin
No. 1	Too cloudy to titrate			157.2	159.9	158.2	170.7	178.4	170.2
No. 2	157.2	158.9	157.9	156.9	158.9	157.5	170.5	176.5	169.5
No. 3	157.7	160.3	160.1	157.3	.....	.....	167.0	174.2	168.3
No. 4	.....	.....	.....	158.8	159.2	158.6	170.9	178.1	169.8
No. 5	156.9	160.3	157.8	154.8	159.1	156.9	173.2	181.2	174.5
No. 6	158.1	159.8	157.9	157.9	159.3	157.8	168.7	174.2	168.2
No. 7	156.7	158.8	157.5	157.0	159.8	157.2	168.2	180.8	170.3
No. 8	155.9	158.5	157.1	155.7	158.5	156.9	171.1	178.8	172.7
Average	157.1	159.4	158.1	157.0	159.2	157.6	170.0	177.8	170.4
Maximum	158.1	160.3	160.1	158.8	159.9	158.6	173.2	181.2	174.5
Minimum	155.9	158.5	157.1	154.8	158.5	156.9	167.0	174.2	168.2
Maximum Deviation	2.2	1.8	3.0	4.0	1.4	1.7	6.2	7.0	6.3
Maximum Deviation from Average	1.2	0.9	2.0	2.2	0.7	1.0	3.2	3.6	3.9
Maximum Deviations in Results Reported									
No. 1	Only averages of nine determinations by three analysts were reported								
No. 2	0.1	0.1	0.6	0.1	1.2	0.3	1.5	2.4	0.9
No. 3	0.1	0.6	0.1	0.2	.....	.....	0.6	0.8	0.5
No. 4	.....	.....	.....	0.5	0.2	0.9	0.6	0.9	0.0
No. 5	0.0	0.0	0.0	0.0	0.3	0.0	0.5	0.5	0.0
No. 6	0.1	0.3	0.4	0.2	0.4	0.1	0.4	0.2	0.4
No. 7	0.1	0.8	1.3	0.3	0.5	0.7	0.6	0.4	0.8
No. 8	0.3	0.0	0.0	0.0	0.0	0.3	0.0	0.0	0.3

The results obtained were regarded by the committee as sufficiently good to warrant the recommendation that the proposed Tentative Methods of Test for Acid Number of Rosin, and Test for Saponification Number of Rosin, appended hereto,[2] be submitted to the Society for publication as tentative. The supporting data on which the methods are based are shown in Table I. In view of the fact that the divergences in the case of saponification numbers are somewhat larger than was expected, the committee feels that another series of cooperative tests should be undertaken on similar samples of rosin before the methods are recommended for adoption as standard.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 505 (1936).

[2] See pp. 835 and 836.

It will be noted that the hot-titration method is recommended for the determination of acid number. The differences obtained between hot and cold titration were insignificant in the judgment of the committee. The hot titration was, therefore, selected since it facilitates dissolving the rosin.

Subcommittee IV proposes to repeat the work on saponification number during the next year, using new cooperative samples. The subcommittee has one other assignment, namely, the development of a method for unsaponifiable matter in rosin. At the present time there seems to be no satisfactory method available. Since it is hardly possible for a committee of this size to work out a method as a committee, it is hoped that some member will find time for the necessary research which the development of such a method requires.

Respectfully submitted on behalf of the subcommittee,

C. C. ZEIGLER,
Chairman.

REPORT OF COMMITTEE D-19
ON
WATER FOR INDUSTRIAL USES

The activities during the year of the several subcommittees of Committee D-19 on Water for Industrial Uses have been directed to the completion of various proposed methods of test. These methods include: (1) General principles governing sampling, prepared by Subcommittee III on Methods of Sampling; and (2) methods for the determination of calcium, magnesium and chloride ions in water, prepared by Subcommittee IV on Methods of Analysis.

Committee D-19 has recommended to the Joint Research Committee on Boiler Feedwater Studies that research investigations be considered for the establishment of a standard method for determining hardness in water that may be either the soap test or an equivalent test, and further that investigations be considered on the development of proposed methods for the determination of hydrogen ion concentration, pH. Committee D-19 has learned that the National Bureau of Standards is initiating a comprehensive and fundamental program on the subject of pH and the committee is accordingly giving consideration to cooperating in this program, with which other interested groups may be associated. In view of the interest in this subject, a paper by a member of the staff of the Bureau of Standards has been secured for presentation at the annual meeting of the Society, as mentioned later in this report.

Committee D-19 is abandoning for the present consideration of methods of testing for zeolite materials. It plans to resume consideration of this matter when more research information is available. Proposed methods on this subject have been published by the American Water Works Association[1] and by the National Lime Association.[2]

On the recommendation of Committee D-19 there was presented to the Society in October, 1936, for publication as information the following proposed methods of analysis of industrial waters:[3]

Proposed Methods for Determination of:

Sulfate Ion in Industrial Water, covering a gravimetric procedure, and, in the appendix, the tetrahydroxyquinone direct titration method. The latter method was edited by Committee D-19 from a report prepared by Subcommittee 8 of the Joint Research Committee on Boiler Feedwater Studies;

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[1] *Journal*, Am. Water Works Assn., Vol. 27, September, 1935, p. 1178.

[2] C. P. Hoover, "Water Supply and Treatment," 2nd Edition, *Bulletin 211*, Nat. Lime Assn., p. 138 (1936).

[3] Available as separate reprint, October, 1936.

Hydroxide Ion in Industrial Water, edited by Committee D-19 from a report prepared by Subcommittee 8 of the Joint Research Committee on Boiler Feedwater Studies, and, in the appendix, the Winkler method, edited by Committee D-19 from a method published in the National Electric Light Association Publication No. 14, "Power Station Chemistry," January, 1931, p. 16;

Total Carbon Dioxide and Calculation of Carbonate and Bicarbonate Ions in Industrial Water, edited by Committee D-19 from a report published by Subcommittee 8 of the Joint Research Committee on Boiler Feedwater Studies, and in the appendix, the barium chloride method edited from Treadwell and Hall, Analytical Chemistry, 6th Edition, Vol. 2, p. 485.

Total Orthophosphate and Calculation of the Respective Orthophosphate Ions in Water. These include a gravimetric procedure and, as an appendix, a volumetric modification of the molybdate method and a colorimetric method, the latter edited by Committee D-19 from a report published by Subcommittee 8 of the Joint Research Committee on Boiler Feedwater Studies.

Committee D-19 is sponsoring a session on water at the 1937 annual meeting of the Society at which the following papers are to be presented:

"The Need for pH Standards," by S. F. Acree, National Bureau of Standards;

"Technique in the Determination of Dissolved Oxygen," by T. H. Daugherty, Hall Laboratories, Inc.;

"A Thermodynamic and Colloidal Interpretation of the Published Studies on the Corrosion Cracking of Stressed Mild Steel in Water Solutions," by J. A. Tajc, Duquesne Light Co.

"Determination of Hardness in Water by Direct Titration," by R. T. Sheen and C. A. Noll, W. H. & L. D. Betz Co.;

"Some Applications of the Polarizing Microscope to Water-Conditioning Problems," by Everett P. Partridge, Hall Laboratories, Inc.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Policy and Procedure (F. N. Speller, chairman).—During the year this subcommittee approved the work now in progress in its several technical subcommittees. No recommendations have been submitted with respect to new developments. On the recommendation of this subcommittee, the question of developing methods of test for zeolites, referred to earlier in this report, is being discontinued for the present.

Subcommittee II, Editorial (E. P. Partridge, chairman).—This subcommittee received no assignment during the current year. It will edit the material to be submitted by Subcommittees III and IV.

Subcommittee III on Methods of Sampling (R. E. Hall, chairman).—This subcommittee has been active during the year in completing that part of its program outlined in the 1936 report[1] relating to (I) Introduction, (II) Scope, and (III) General principles governing sampling. The subcommittee expects to refer this part of the report to Committee D-19 this year for submission to the Society for publication. Work will be done by the subcommittee on item IV, Sampling water in the liquid state.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 508 (1936).

Subcommittee IV on Methods of Analysis (J. R. McDermet, chairman).—This subcommittee has been active during the year in completing proposed methods for the determination of calcium, magnesium and chloride ions in water. It expects to refer these proposed methods to Committee D-19 for submission to the Society for publication. Subcommittee IV plans to undertake the preparation of proposed methods for the determination of silica and dissolved oxygen in water.

This report has been submitted to letter ballot of the committee which consists of 49 members; 34 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

R. E. HALL,
Temporary Secretary.

REPORT OF COMMITTEE E-1
ON
METHODS OF TESTING

Committee E-1 on Methods of Testing held a meeting in Atlantic City, N. J., on July 2, 1936, subsequent to the presentation of its 1936 report to the Society.

Consideration has been given during the year to a review of the personnels of the technical committees and sections of Committee E-1 and it is now felt that the various standing committees of the Society are in general adequately represented on those subcommittees whose work is of direct interest.

The Executive Committee of the Society appointed three representatives-at-large to serve on the Advisory Committee, namely, H. F. Gonnerman, Portland Cement Assn., H. H. Morgan, Robert W. Hunt Co., and J. R. Townsend, Bell Telephone Laboratories, Inc., each for three-year terms, expiring in 1939.

In October, 1934, there was referred to the standing committees of the Society by Committee E-1 the question of the desirability of having a study made looking toward the establishment of standard atmospheric test conditions, including both humidity and temperature. As a result of the interest expressed in the subject, a meeting of representatives of the interested standing committees was held on July 1, 1936, under the chairmanship of Mr. Robert Burns of the Bell Telephone Laboratories. The conference resulted in the recommendation to Committee E-1 that it organize a coordinating committee with the following functions: (1) to promote standardization by encouraging the adoption of existing methods and by minimizing so far as possible the number of required new methods; (2) to provide a clearing house for information obtained by standing committees; and (3) to furnish educational and advisory service to the Society in particular and to industry in general. It was also recommended that the proposed subcommittee consider, as one of its first activities, the adoption of a definition for conditioning. The Advisory Committee of Committee E-1 will give further consideration to this subject at a meeting to be held during this annual meeting.

Subsequent to the 1936 annual meeting, Committee E-1 presented to the Society through Committee E-10 on Standards a proposed Tentative Method of Test for Softening Point by Ring-and-Ball Apparatus (E 28 –

36 T). The new tentative method was accepted[1] by Committee E-10 on August 26, 1936, and appears in the 1936 *Proceedings*.[2]

ACTIVITIES OF TECHNICAL COMMITTEES

Technical Committee I on Mechanical Testing (H. F. Moore, chairman):

As is usual the report of this technical committee consists of very brief summaries covering the work of its various sections during the past year.

Section on Tension Testing (R. L. Templin, chairman).—This section held a meeting on March 3, 1937, in Chicago, Ill., during the spring group meetings of A.S.T.M. committees. At that meeting it was reported that the discussion with Committee A-1 on Steel in regard to changes in the Standard Methods of Tension Testing of Metallic Materials (E 8 – 36) was still being carried on, and that certain questions regarding the testing of specimens of steel pipes were being given special attention. It was reported that the revisions of Standard Methods E 8, which were required in order to bring the methods into agreement with the Standard Definitions of Terms Relating to Methods of Testing (E 6 – 36), had been incorporated in the methods as last printed.[3]

Previous to this meeting the secretary of Committee E-1 prepared for the section a statement covering various provisions for determining tensile strength appearing in A.S.T.M. specifications for metals. These provisions were discussed in considerable detail and it was suggested that the attention of the standing committees concerned should be directed to the provisions of the Standard Methods of Tension Testing of Metallic Materials (E 8 – 36) with the idea of making the product specifications agree with the Standard Methods E 8 as closely as may be feasible. If the committees responsible for the materials specifications have any reasons for not making them agree with Standard Methods E 8, it is hoped that those reasons will be brought to the attention of the Section on Tension Testing for consideration in connection with the subsequent revision of Methods E 8 – 36.

Very few criticisms or suggestions regarding Methods E 8 – 36 have been received and it was concluded, therefore, that they are in satisfactory form.

Section on Compression Testing (M. F. Sayre, chairman) and *Section on Effect of Speed of Testing* (P. G. McVetty, chairman).—The work of these two sections is considered together because the Section on Compression Testing has found that any further steps toward standardization of methods of compression testing are closely related to the question of effect of speed of testing. These sections plan to hold meetings during the annual meeting in June.

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[1] In submitting this tentative method to Committee E-10 on Standards, Committee E-1 reported results of the letter ballot vote as follows: Of a total membership of 52, 46 members returned their ballots, of whom 29 voted affirmatively, 1 negatively, and 16 members marked their ballots "not voting."

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1186 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 1300.

[3] See 1936 Book of A.S.T.M. Standards, Part I, p. 833.

It is to be noted that last year the paper on "The Effect of Testing Speed on Strength and Elastic Properties of Concrete" by Paul G. Jones and F. E. Richart[1] described a test method, by which this problem of effect of speed could be studied. It is hoped that it will be possible to arrange for a similar study of the effect of speed of testing on metals, and the matter of compression tests can then be taken up. For metal structures under steady compressive load the yield strength of the metal seems to be a very important property. Compression tests for yield strength can be easily carried out and the question of the use of compression tests for yield strength seems worthy of consideration.

Section on Bend Tests for Ductility (A. B. Kinzel, chairman).—The work of reassembling the test data obtained by the Section on Bend Tests, which were lost by fire, is still progressing and contact is being maintained between this section and the Section on Testing Thin Sheet Metals. The section plans to hold a meeting during the annual meeting of the Society in June. It is hoped that the final report on cupping tests for ductility will be acted on at that meeting.

Section on Indentation Hardness (J. R. Townsend, chairman).—This section is planning to hold a meeting during the annual meeting in June. The Methods of Rockwell Hardness Testing of Metallic Materials (E 18 - 36) were adopted as standard last year and no further revisions are contemplated at this time. The section is sponsoring a paper by R. H. Heyer on "Analysis of the Brinell Hardness Test,"[2] which is being presented at this annual meeting of the Society.

At the approaching meeting, the section proposes to discuss some general problems, such as the limitation of thickness of sheet metal which can be satisfactorily tested by the Rockwell hardness tester.

Section on Testing Thin Sheet Metals (C. H. Marshall, chairman).—In March, 1934, this section began the collection of information regarding various tests as applied to sheet metals, the object of this work being to obtain data which would be of use in the activities of the section and to obtain information which should be of assistance in developing standards for testing sheet metals. The chairman of the section has completed the draft of a proposed report on "Usefulness and Limitation of Various Tests as Applied to Sheet Metals," which is a condensed summary of several more-detailed draft outlines prepared during the progress of the work. The report has been approved by the members of the section and is presented in Appendix I.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 380 (1936).
[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 119 (1937).

Section on Impact Testing (W. W. Werring, chairman).—This section is planning cooperation with Subcommittee IX on Steel Tubing and Pipe of Committee A-1 on Steel and with the Committee on Pipe of the Association of American Steel Manufacturers Technical Committees, in a study of tension-impact testing of tubular steel products.

Proposals have been received from several sources for the addition of keyhole notch specimens to the Tentative Methods of Impact Testing of Metallic Materials (E 23 – 34 T). This question will be considered at the next meeting of the section.

The section is planning, for a later date, a symposium on impact testing, which is to be held under the joint auspices of the A.S.T.M. and the Committee on Industrial Welding Research of the Engineering Foundation, with the cooperation of the American Society of Mechanical Engineers. Prof. M. F. Sayre is officially acting as chairman of the Subcommittee on Methods of Testing of the Committee on Industrial Welding Research. In preparation for this symposium the section will hold a Round-Table Discussion on Impact Testing on June 30 during this annual meeting of the Society. Presentation of problems and discussion of the major gaps in the information on impact is to be encouraged at the round-table meeting in an effort to lay the foundation for an informative, coordinated symposium, to be held possibly in 1938. The section has planned the round-table discussion as a compact informal meeting in the hope of stimulating discussion.

Section on Elastic Strength of Materials (F. B. Seely, chairman).—This section held a meeting on March 3, 1937, in Chicago. At that meeting a report was presented by Col. G. F. Jenks of the Ordnance Department, U. S. Army (formerly of Watertown Arsenal), on "Yield Strengths Corresponding to Small Percentages of Set." The results of this investigation are presented as information in Appendix II to this report.

At the March meeting, a committee was appointed to collect a number of representative stress-strain diagrams of the different metals covered by A.S.T.M. specifications. It is hoped to use this collection as a basis for the study of the question of reporting values of yield strength for materials. Of especial interest is the determination of yield strength for a material which has a stress-strain diagram that is curved all the way from zero stress.

Section on Calibration of Testing Machines and Apparatus (H. F. Moore, chairman).—This section held a meeting on March 3, 1937, in Chicago. At that meeting a preliminary outline for the consideration of the problem of calibrating strain apparatus was presented by Mr. B. L. Wilson of the National Bureau of Standards. The main item of the discussion was in connection with the work of Committee D-13 on Textile Materials. The question given special consideration was that of calibrating testing machines for textiles and rubber "at speed." After considerable discussion it was the opinion of the section that the direct calibrating of machines at speed was less accurate and less reliable than the calibration of such machines "statically" by means of dead weights, proving lever, or elastic devices, with computed allowances for the inertia effects when the machine was used at various speeds. It was noted that these inertia effects do not depend directly upon the speed of the *pulling head* but can be calculated if there is available a load-time diagram which is representative for a given type of material and a given size of specimen. This viewpoint was brought to the attention of the chairman of Committee D-13, for consideration by the committee.

The section also considered the possibility of using a standardized single lever apparatus, or a frequently standardized beam-balance testing machine as a primary standard for the calibration of elastic devices. No action was taken on this subject.

Technical Committee II on Consistency, Plasticity, Etc. (E. C. Bingham, chairman):

The technical committee has been enlarged to include several adviser-members from both this country and abroad and also representatives from other technical societies. In addition to those mentioned in the report of the committee last year,[1] Mr. G. G. Sward has been added to the committee as the representative of the National Paint, Varnish and Lacquer Assn.

A meeting of the technical committee was held in Atlantic City on July 1, 1936, at which meeting it was decided that the time was propitious for a formal presentation of a series of papers on the subject of consistency. The committee is accordingly sponsoring a Symposium on Consistency at this annual meeting of the Society.

The World Petroleum Congress at its meeting in London in 1936 took action advocating that all viscosities be expressed in absolute units. It appears that there is apparent unanimity of opinion on this matter but conditions in practice are quite to the contrary. Where properties such as plasticity, consistency or hardness are measured, the result is normally obtained as a *single* numerical quantity although it must in some way represent two or more independent variables. So long as industry tolerates the present lack of acceptable definitions, standards, and methods of measurement, this very deplorable condition will continue. The word "deplorable" is used advisedly because, although the scientist has long recognized that the above situation is absurd, these properties of hardness, tensile strength, etc., with or without proper characterization, continue to be used in the most important and far-reaching questions of control. It is for these reasons, that the committee has arranged for a second symposium on the subject of consistency. The purpose of the first series of papers on this subject, held in 1923,[2] was to ascertain what consistency is. It is proposed to consider in the present symposium the advances made in consistency measurement, including a discussion of the theoretical background and the existing nomenclature, the possibilities of standardization, and the use and advantages to be derived from the use of absolute units of measurement. The papers comprising the symposium are as follows:[1]

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[1] *Proceedings,* Am. Soc. Testing Mats., Vol. 36, Part I, p. 521 (1936).
[2] *Proceedings,* Am. Soc. Testing Mats., Vol. 23, Part II, p. 432 (1923).

"Introduction," by W. H. Fulweiler, Consulting Chemist,

"Recent Progress in Consistency Measurement," by E. C. Bingham, Lafayette College,

"Definition of Consistency and Theoretical Considerations," by Melvin Mooney, U. S. Rubber Products, Inc.,

"Consistency Measurements in the Paint Industry," by D. L. Gamble, The New Jersey Zinc Co.,

"The Flow Properties of Asphalts Measured in Absolute Units," by R. N. Traxler, The Barber Co., Inc.,

"Consistency Measurements in the Coal Tar Industry," by E. O. Rhodes, E. W. Volkmann and C. T. Barker, Koppers Products Co.,

"Viscosity Measurement of Petroleum Products and Lubricants," by J. C. Geniesse, The Atlantic Refining Co.,

"Consistency Measurement of Rubber and Rubber Compounds," by J. H. Dillon and L. V. Cooper, The Firestone Tire and Rubber Co.,

"Measurements of Flow Characteristics of Thermosetting Resins," by H. L. Bender, H. F. Wakefield and H. E. Riley, Bakelite Corp.

"Cold Flow of Insulating Materials," by Robert Burns and Irving L. Hopkins, Bell Telephone Laboratories, Inc.

The Special Committee on Softening Point Test (H. M. Hancock, chairman) completed the development of a Method of Test for Softening Point by Ring-and-Ball Apparatus (E 28 – 36 T) which was accepted by the Society for publication as tentative, as mentioned earlier in this report. The new method was intended as a revision to replace eventually the present Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (D 36 – 28), under the jurisdiction of Committee D-4 on Road and Paving Materials. Until certain criticisms of the new method under consideration by Committee D-4 have been investigated, the present Standard D 36 is being continued as a standard of the Society.

Suggestions for revision of Tentative Method E 28, including revised requirements for the high-softening-point thermometer and a proposed method for preparing the ring-and-ball softening point test specimens by use of a casting plate, will be considered by Technical Committee II at its meeting to be held during the annual meeting in June. Proposed revisions of the method may be offered to the Society during the summer through Committee E-10 on Standards.

Criticisms of the Tentative Definitions of Terms Consistency and

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[1] See "Symposium on Consistency: Critical Discussion of Present-Day Practices in Consistency Measurements," December, 1937. (Available as separate publication.)

Plasticity (E 24 – 34 T) received during the year will be considered in detail at the meeting of the committee to be held in June.[1]

Technical Committee III on Particle Size and Shape (L. T. Work, chairman):

The standardization of sieves has been delegated to the Sectional Committee on Sieves for Testing Purposes (A.S.A. Project: Z 23) which is functioning under the joint sponsorship of the National Bureau of Standards and the A.S.T.M., under the procedure of the American Standards Association. The work of the sectional committee is closely related to the work of Technical Committee III.

After considerable deliberation, proposed Specifications for Sieves for Testing Purposes, which will cover coarse and fine sieves, have been developed and have received the favorable vote of practically the entire membership of Sectional Committee Z 23. The proposed specifications represent a revision of the existing A.S.T.M. Standard Specifications for Sieves for Testing Purposes (E 11 – 26), and for Round-Hole Screens for Testing Purposes (E 17 – 36). The revision of the specifications is minor in character with the exception of one feature concerning which some question has arisen, namely, the specification of wire diameter. The new specifications propose a range of wire diameter rather than a stated diameter. There are other revisions of minor importance and there has been a correlation of the fine and coarse sieves so that the break at four mesh no longer exists. It has been found feasible to continue the fourth root of two into the coarser sizes and to adopt the greater number of fractional inch sieves in that series. Where the deviations are outside of the tolerance limits, two cloths have been specified in order to emphasize that they are not interchangeable. It is expected that the proposed specifications will be referred to Technical Committee III shortly.

Section on Subsieve Sizes (D. L. Gamble, chairman).—A meeting of a group interested in the revision of the present Tentative Method of Analysis for the Particle Size Distribution of Subsieve Size Particulate Substances (E 20 – 33 T) from the standpoint of its adaptation to pigments was held at Columbia University, New York City, on February 15, 1937.

On the basis of suggestions made at this meeting and with the cooperation of individual members of the group, the chairman is in the process of revising the tentative method for reconsideration by this group. It is the intention of the section to consider eventually similar revisions from the standpoint of other subsieve size particulate substances.

Technical Committee IV on Methods for Density (H. W. Bearce, chairman):

The various methods of test for density and specific gravity prescribed in the standards of the Society are being reviewed by the chairman for the purpose of preparing recommended changes and improvements in the interest of having all descriptions of such tests theoretically and basically correct.

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[1] Subsequent to the annual meeting Committee E-1 presented to the Society through Committee E-10 on Standards revisions in these definitions, see Editoral Note, p. 517.—ED.

Technical Committee VII on Chemical Composition (G. E. F. Lundell, chairman):

Technical Committee VII has held no meetings during the past year. Numerous questions concerning methods of chemical analysis have been raised in connection with the chemical requirements inserted in specifications that are being prepared by various committees, chiefly in the ferrous and non-ferrous groups. Most of these questions have, therefore, been referred for action to Committee E-3 on Chemical Analysis of Metals.

An interesting development during the year has been the tendency on the part of some committees to insert "precision" limits in methods for the chemical analysis of metals. The failure of the committees to distinguish between precision and accuracy emphasizes the need for the round-table discussion on the subject which is to be held at this annual meeting of the Society, as mentioned later in this report.

Technical Committee IX on Interpretation and Presentation of Data (H. F. Dodge, chairman):

This committee's Manual on Presentation of Data has had a wide distribution since it was originally issued in 1933.[1] Two Supplements, *A* and *B*, covering additional material were subsequently prepared at the request of the committees of the Society and separately issued in 1935.[2]

Early in 1937, the supply of the second printing of the Manual was exhausted. Use of the Manual and its two Supplements had indicated the desirability of combining this material in one volume and amplifying certain sections of the text. The new printing was accordingly set up in this manner and made available in pamphlet form in March, 1937. The committee took the opportunity of making a number of minor changes in text wording and, on the basis of specific requests made at the last annual meeting, amplified two sections of the Manual. The first of these related to short-cut methods of computing averages and standard deviations and there has been added what is termed "Short Method No. 2," involving a succession of cumulative sums whereby constants are determined by simple additions, a convenience when no multiplying computing machine is available. The second amplification was incorporated in Supplement A and consisted in adding a set of curves to assist in determining constants needed for various "chances" associated with the ± limits of uncertainty of an observed average, for example, average = 575.2 ± 5.1 lb.

During the year the American Statistical Association, signifying its interest in the work of the committee, suggested that some means be set up for coordinating this type of work in the two Societies and requested that a short article on the committee's activities be prepared for publication in its journal. Such an article was prepared by the chairman of this technical committee and published in the June, 1937, issue of the *Journal of the American Statistical Association.*

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 451 (1933).

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, pp. 1411 and 1418 (1935).

Arrangements have been made for conducting a round-table discussion on the subject of "Precision and Accuracy," to be held jointly with Technical Committee XI on Designation and Interpretation of Numerical Requirements, at this annual meeting of the Society.[1] The three principal speakers for the round-table discussion are: Messrs. W. A. Shewhart, G. E. F. Lundell and H. F. Moore.

During the year, the chairman, H. F. Dodge, presented a paper on "Statistical Methods and Specifications of Quality," at a meeting sponsored by the Cleveland A.S.T.M. District Committee and the Cleveland Section, A.S.M.E., at Cleveland, Ohio, on January 28, 1937. The paper was published in the A.S.T.M. *Bulletin,* March, 1937.

The committee has continued to assist other committees of the Society in connection with specific problems dealing with statistical methods and the analysis of data.

Technical Committee XI on Designation and Interpretation of Numerical Requirements (J. A. Gann, chairman):

The principal activity of this technical committee during the year has been devoted to furthering consideration by the standing committees of the Society to the proposed Recommended Practices for Designation of Numerical Requirements in Standards, published as information in an appendix to the 1936 report[2] of the committee. In order to show the manner of applying the recommendations for the designation of numerical requirements in standards in accordance with the proposed recommended practices, the committee prepared two illustrative specifications. These have been appended to reprint copies of the proposed recommended practices and are being made available to the committees of the Society for detailed consideration.

As mentioned earlier in the report, this technical committee is sponsoring at this annual meeting of the Society, in cooperation with Technical Committee IX on Interpretation and Presentation of Data, a Round-Table Discussion on Precision and Accuracy.

This report has been submitted to letter ballot of the committee which consists of 52 members; 33 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

P. J. SMITH,
Ex-officio Secretary.

W. H. FULWEILER,
Chairman.

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[1] See Summary of Proceedings, p. 3.
[2] *Proceedings,* Am. Soc. Testing Mats., Vol. 36, Part I, p. 527 (1936).

EDITORIAL NOTE

Subsequent to the annual meeting Committee E-1 on Methods of Testing presented to the Society through Committee E-10 on Standards a proposed revision of the Tentative Definitions of Terms Consistency and Plasticity (E 24 – 34 T) in the form of new Definitions of Terms Relating to Rheological Properties of Matter. These definitions were accepted by Committee E-10 on October 11, 1937, and appear on p. 1300.

APPENDIX I

REPORT ON USEFULNESS AND LIMITATION OF VARIOUS TESTS AS APPLIED TO SHEET METALS

In March, 1934, the Section on Testing Thin Sheet Metals began the collection of information regarding various tests as applied to sheet metals, the object of this work being to obtain data which would be of use in the activities of the section and to obtain information which should be of assistance in developing standards for sheet metals testing. It was soon found that the desired information could best be obtained by means of a questionnaire which has resulted in the collection of a great amount of useful information from many persons engaged in various phases of sheet metals testing so that it can be used for drawing definite conclusions regarding the usefulness and limitations of various tests for sheet metals.

In this report, the statements given are based on the information obtained regarding the usefulness and limitation of various tests applied to sheet metals. Consideration has also been given to information obtained through the several round-table discussions on various phases of sheet metals testing[1] which were sponsored by the section, and from other sources.

There is presented in Table I a summary of the replies from the 56 laboratories that cooperated with the committee in furnishing the information which forms the basis for this report.

Table I.—Summary of Replies Received from Fifty-six Cooperating Laboratories.

Test	Used	Used for Inspection	Used for Research	Considered Most Valuable	Used Only Occasionally	Considered Unreliable or Not Useful (for Their Purpose)
Tension	50	29	25	34	12	..
Microscopic	40	23	23	19	10	..
Cupping	46	19	16	17	9	2
Hardness:						
Brinell	23	12	11	8	6	3
Rockwell	43	24	19	28	6	3
Vickers	9	4	10	8	2	..
Monotron	5	..	2	1	..	..
Scleroscope	20	9	9	3	9	11
Other Hardness Tests	4	..	..	..	1	..
Cold Bend	42	24	15	12	8	..
Fatigue	11	..	13	..	3	1
Shear or Punching	8	4	6	..	2	1
Impact	8	1	8	..	..	..
Creep	5	1	7	2	..	..
Other Tests	6	1	3	2	1	..

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[1] During the past several years the section sponsored three round-table discussions, the first on "Use of the Tension Test for Judging the Suitability of Sheet Materials for Various Purposes," held in Chicago, Ill., at the 1933 annual meeting, see *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 563 (1934); the second on "Hardness Testing of Sheet Metals," held in Atlantic City, N. J., at the 1934 annual meeting; and the third on "Ductility Testing of Thin Sheet Metals," held in Detroit, Mich., at the 1935 annual meeting.

Tension Test:

The tension test is without doubt the most used and most valuable test for sheet metals. It is used in its simplest form, with measurement of ultimate strength and elongation, for control and acceptance tests of all materials.

With the use of extensometers of various kinds and the determination of various elastic or plastic properties, tension tests are used by research laboratories in determining fundamental information regarding various materials and their suitability for various applications.

Frequently, elastic properties are determined in connection with tension tests made primarily for control or acceptance.

Sometimes, tension tests on samples cut from sheet in various directions with respect to the direction of rolling give useful information as, for example, in determining the suitability for deep drawing operations.

Several laboratories have reported difficulty in testing very thin sheet metals and consider the tension test unreliable on materials of the order of 0.005 in. in thickness or less. Other laboratories have reported the use of tension tests for much thinner materials. It is evident that success in testing very thin materials is dependent upon having suitable apparatus and skillful manipulation, granting which, there is probably no limit to the thinness of materials to which the tension test can be applied. It is, of course, evident that for thin materials unusual precautions must be taken in obtaining accurate measurements of the thickness.

Microscopic Tests:

Second only in use and importance to the tension test, microscopic tests both for surface appearance and for determination of grain size are used and considered extremely valuable for both ferrous and non-ferrous sheet metals.

Cupping Tests:

For both inspection and research, cupping tests are considered valuable and are quite generally used.

It is generally recognized that considerable judgment must be used in the making and interpretation of these tests as the amount of cupping to which material may be subjected before failure is of less value in predicting its behavior in service or under stamping forming operations than the appearance of the dome or cup formed during the test.

It usually is considered that a circular depression around the formed cup near the dome indicates material which has approximately equal strength in all directions, while splitting on one side or even across the dome indicates materials which are definitely weaker or of less ductility in the direction of the split.

This test also is used to a considerable extent as an indication of grain size, which can be judged by the appearance of the stretched metal of the dome. A smooth dome indicates a fine grain material while a rough dome is an indication of large grain size in the material tested.

Hardness Tests:

Brinell.—The Brinell hardness test is used by many laboratories, particularly for materials of the order of $\frac{1}{8}$ in. or thicker. The "baby" Brinell with $\frac{1}{16}$-in. ball is used to some extent for thin materials but usually for investigation or research due to the time involved in preparing samples for and in making these tests.

From the reports received, it is evident that Brinell and other indentation hardness tests are frequently used for materials which are so thin that misleading results are likely to be obtained.

Rockwell.—The Rockwell hardness test is the most generally used of all the indentation hardness tests, both for control and research.

Since the smooth parallel surfaces necessary to the success of this test are not usually difficult to obtain in sheet metals it is well adapted for testing these materials.

The recent development of the "superficial" Rockwell hardness tester has extended this test to much thinner sheet materials than formerly could have been successfully tested by this method.

While no absolute rule has as yet been arrived at, it is an accepted fact that where a mark is produced on the surface in contact with the anvil, results should be considered as possibly not representing the actual hardness of the materials.

For very thin sheet metals, Rockwell hardness tests are sometimes used, for comparison only, with others of the same kind and thickness. If this is done, consideration must be given to the effect of various kinds of anvils, presence or absence of oil or other materials on the anvil or test specimen, and it must also be borne in mind that when the penetration is deep with respect to thickness there may be an apparent *decrease* of hardness due to "flow" or "punching through" of the material due to lack of lateral support. For thinner material where penetration is even greater with respect to thickness, pressure of the penetrator on the anvil may result in an apparent increase in hardness.

Vickers.—The Vickers hardness test is used to some extent both for control and research. Apparently this test ranks with the Rockwell test in respect to usefulness but has not come into nearly so general use.

Monotron.—This test is reported as useful by many laboratories but apparently has not been generally adopted and very little specific information has been obtained regarding it.

Scleroscope.—The scleroscope is used to a considerable extent for testing sheet metals, and useful information is obtained through its use.

It is evident from the comparatively large number of persons who consider this test unreliable or not useful for sheet metals that extreme care and good judgment must be used when it is applied, particularly in the case of the "softer" materials.

For very thin metals, the same considerations as have already been noted under Rockwell tests undoubtedly apply and the same precautions should be taken not to compare tests on any particular thickness of material with either dissimilar materials of even the same thickness or with any materials of different thicknesses.

Cold Bend Tests:

Under this classification are considered only tests in which the materials are bent either a definite distance, such as 45 deg., 90 deg. or 180 deg. around a fixed radius, or flat on itself, the appearance or condition afterward indicating the quality or suitability for some definite use.

Tests of these kinds are extensively used for all kinds and tempers of sheet and strip materials. They are generally considered useful in giving information of practical value.

Fatigue Tests:

The usefulness of the fatigue test is apparently confined to research and investigation. Various methods of making tests are reported and it apparently is used mostly for testing spring materials.

Shear or Punching Tests:

While tests for shear or punching are used to some extent, particularly for inspection, reports from various laboratories indicate that, in general, little information of value with regard to sheet metals is obtained by such tests.

Impact Tests:

Impact tests may not usually be thought of in connection with sheet metals, but several laboratories have reported that impact tests are used as a research test for obtaining information on sheet metals.

Creep Tests:

Creep tests are used entirely for research but to a considerable extent for indicating rate of flow or stretching of material under continuous load.

Stiffness Tests:

Tests for stiffness are used to some extent, chiefly for determining either elastic or bending properties (dependent on modulus of elasticity) of materials to be used for springs or when information regarding the "springiness" is of importance.

Corrosion, Electrical, Sag, and Other Special Tests:

Special tests have been reported as being used to give information regarding suitability of materials intended for specific applications.

Respectfully submitted on behalf of the Section on Testing Thin Sheet Metals,

C. H. MARSHALL,
Chairman.

APPENDIX II

YIELD STRENGTHS CORRESPONDING TO SMALL PERCENTAGES OF SET

These studies were undertaken to determine whether the methods recommended for determining yield strength as given in the Standard Definitions of Terms Relating to Methods of Testing (E 6 – 36)[1] should include some method of determining yield strength corresponding to a very small amount of offset (of the order of 0.003 per cent) for which the existing methods may not be satisfactory.

Typical ferrous and non-ferrous metals and alloys were tested and the yield strengths were measured by various methods at the Watertown Arsenal.[2] The work was done on an Amsler testing machine, using a Baldwin-Southwark autographic-recorder.

Ferrous Metals:

The typical steels tested were S.A.E. No. 2340, heat treated, and S.A.E. No. 4150, as rolled. In the first, the difference between the proportional elastic limit (0.000 per cent offset) is 3000 lb., or about 4 per cent of the yield point. This difference is much lower than in many heat-treated steels tested, as, for example, in a nickel-steel forging as indicated in Table I. In this latter case, the heat-treated forging approaches a condition of which the S.A.E. No. 4150 as-rolled steel is typical. The shape of the stress-strain curve just below the yield point is typical of metal containing internal stresses. These stresses may be removed by aging or stabilization heat treatments, and the proportional elastic limit elevated to just under the yield point as normally determined.

The inclusion of a requirement for a very small percentage offset for such steels would result in more careful processing in order to obtain full advantage of any chemical composition. It is justified only for the more carefully processed materials, such as Ordnance steels, because of costs involved.

An offset of 0.003 per cent is too small to obtain by careful measurements in a first class testing laboratory. For the most careful work a value of 0.005 per cent would appear to be the smallest practicable. Such a value would be justified only for high-quality quenched-and-drawn steels.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 861, and Part II, p. 1429.
[2] Watertown Arsenal *Report No. 111.2–11.*

In the case of the S.A.E. No. 4150 steel, as rolled, the difference between the 0.000 per cent set and the yield point (dividers method) is much greater, being about 28 per cent of the yield point value. Similar results would have been obtained on a carbon steel in the as-rolled condition. The method of processing and the use to which such materials are put does not warrant methods of testing being more refined than are now provided for in A.S.T.M. standards.

Non-Ferrous Metals:

Non-ferrous materials normally have types of stress-strain curves as found in the case of hard bronze (see Fig. 1). In the testing of such materials, the use of abnormally small offset percentages has no place.

TABLE I.—RESULTS OF TESTS FOR YIELD STRENGTH.

Figure	Material	Yield Strength, lb. per sq. in.							Proportional Limit, Army Method, lb. per sq. in.	Yield Point by Dividers, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent
		Offset (from modulus line), per cent											
		0.000	0.003	0.010	0.020	0.050	0.100	0.200					
1	S.A.E. No. 2340, heat treated...	67 000	68 000	69 500	69 750	69 750	69 500	70 000	69 250	70 000	107 500	25.0	54.1
1	Bronze, hard....	24 500	28 000	32 000	35 500	45 500	55 000	65 500	55 000	53 000	94 500	19.3	67.3
2	S.A.E. No. 4150, as rolled......	32 000	36 000	40 000	42 500	44 250	44 250	.....	41 750	44 250	85 500	31.5	69.2
2	Stainless steel, 18 per cent chromium, 8 per cent nickel.........	14 750	17 000	20 000	22 750	26 000	28 250	.....	22 000	31 000	83 000	63.6	76.0
3	Stainless iron, as rolled.........	92 000	97 000	102 000	108 000	115 500	.....	.....	113 000	......	171 000	15.7	54.8
3	Stainless iron, after 650 C. draw..........	53 000	55 500	60 000	63 500	68 500	72 000	.....	64 500	74 000	94 500	22.9	71.0
4	Monel metal, as rolled.........	38 000	40 000	45 500	50 250	55 000	58 000	.....	54 000	61 750	85 500	32.9	67.0
4	Monel metal, 300 C. draw.......	49 000	50 500	53 500	55 250	58 750	60 500	.....	57 000	62 000	86 000	34.3	66.3
4	Monel metal, 550 C. draw.......	51 500	53 000	54 500	55 750	57 500	58 750	.....	55 500	61 000	87 500	35.0	65.4
5	Nickel-steel forging, heat treated	57 000	63 000	73 000	81 000	91 500	95 000	96 500	90 500	96 500	120 000	21.0	56.3

An interesting family of curves are those for monel metal, as indicated in Fig. 4. The requirement for yield strength (0.01 per cent or 0.02 per cent offset) equaling 50,000 lb., or over, would have necessitated an additional heat treatment with corresponding additional cost. Such expense is justified only under exceptional circumstances.

Conclusions:

Very small values of offset are required only for heat-treated steels of high quality and possibly some other materials in which design requirements are severe.

An offset percentage of less than 0.005 per cent is not justified except for experimental purposes.

There is no need for a revision of the methods for determining yield strength as recommended in the A.S.T.M. Standard Definitions of Terms Relating to Methods of Testing (E 6 – 36) in case it is desirable to use such small offset values.

A.S.T.M. specifications for metals do not cover materials in which a very low percentage offset is required. The methods for determining yield strength as recommended in the Standard Definitions E 6 should, however, be of general application and be suitable for use in the case of specifications covering special quality materials. The existing methods are considered to be sufficiently general for such use. The special specification should give the offset percentage value to be used. It is considered that these A.S.T.M. methods are not intended to apply and should not apply to special experimental work.

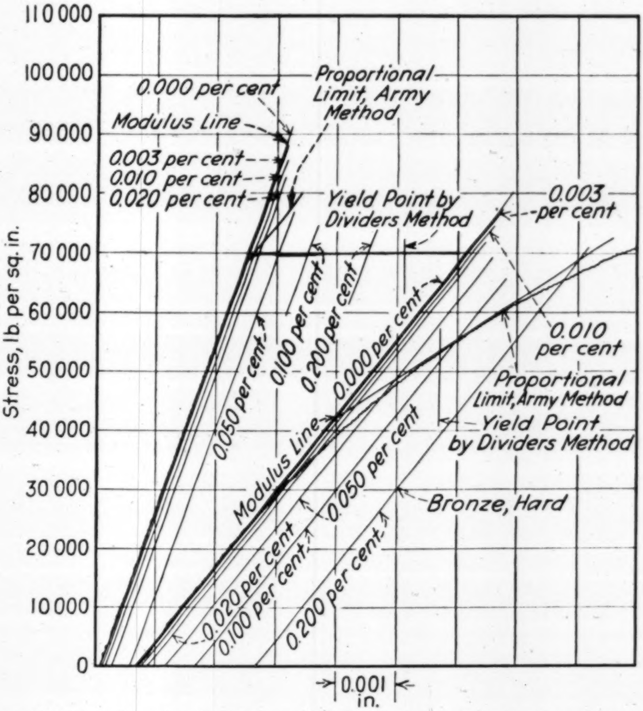

FIG. 1.—Yield Strengths of S.A.E. No. 2340 Steel (Heat Treated), and Hard Bronze.

Respectfully submitted on behalf of the Section on Elastic Strength of Materials,

G. F. JENKS.

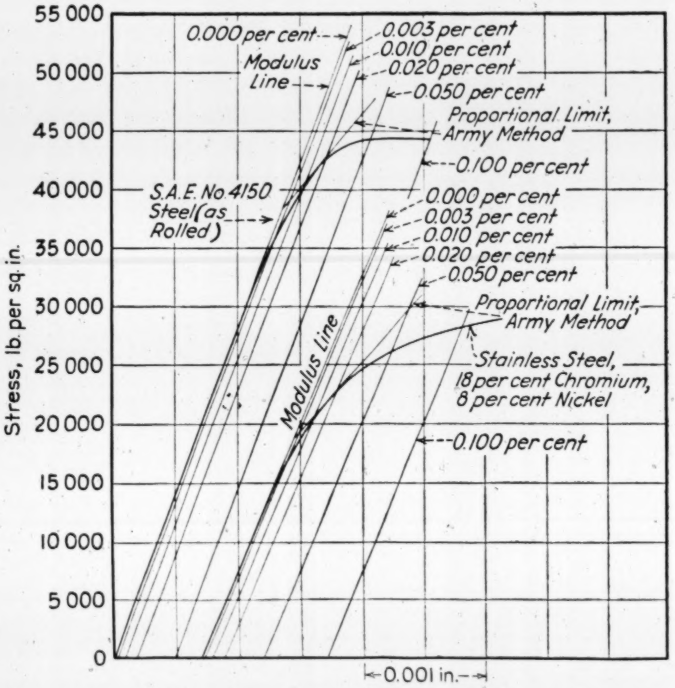

FIG. 2.—Yield Strengths of S.A.E. No. 4150 Steel (as Rolled), and an 18 per cent Chromium, 8 per cent Nickel Steel.

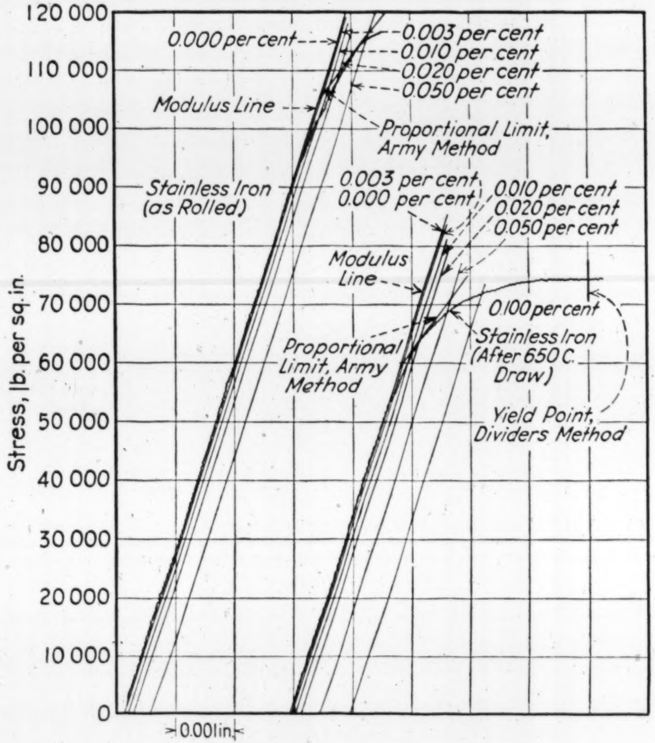

FIG. 3.—Yield Strengths of Stainless Iron.

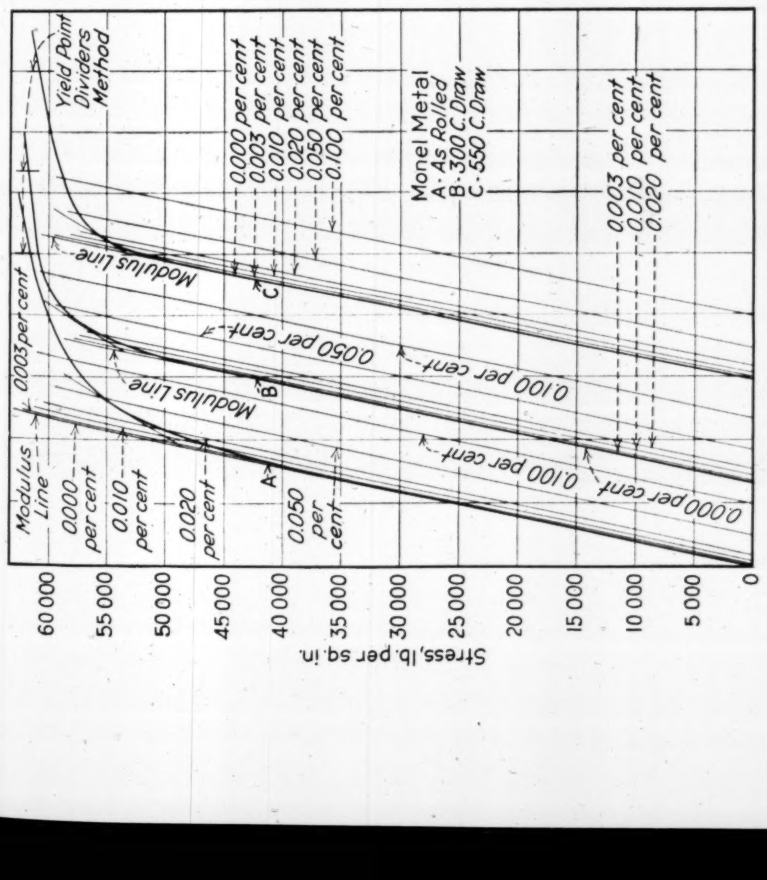

FIG. 4.—Yield Strengths of Monel Metal.

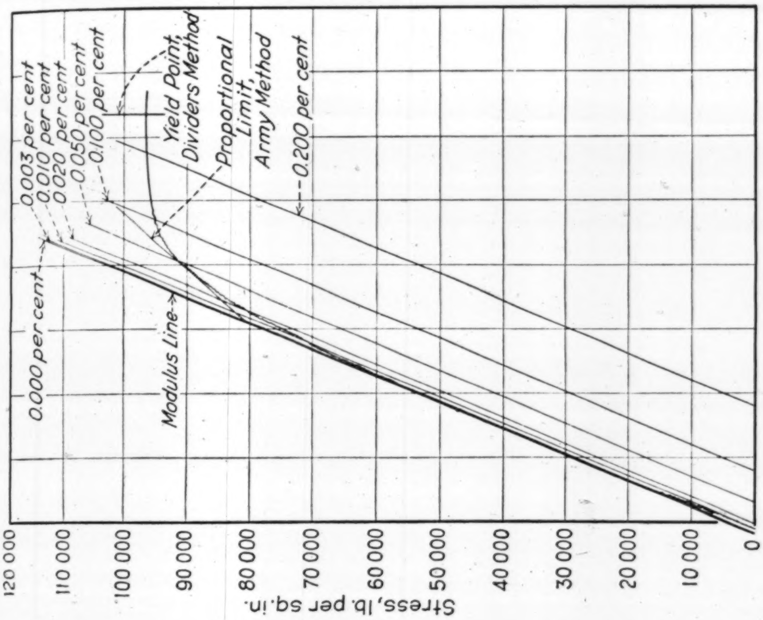

FIG. 5.—Yield Strengths of Nickel-Steel Forging (Heat Treated).

REPORT OF COMMITTEE E-2
ON
SPECTROGRAPHIC ANALYSIS

Committee E-2 on Spectrographic Analysis has held no meetings since its last meeting held at the time of the 1935 annual meeting in Atlantic City, N. J. Subcommittees have continued the work begun in previous years on the formulation of methods of analysis and on the development of sources of metals of extreme purity, so urgently needed as a base for the preparation of standards for quantitative spectrographic analysis.

A special subcommittee under the chairmanship of Thomas A. Wright, has drawn up a set of regulations governing Committee E-2. These were discussed and approved by the committee at its June meeting for submission to letter ballot.

The committee is attempting to arrange for immediate and future periodic publication of an extensive Bibliography on Spectroscopy which had been assembled and is being kept up-to-date by Mr. W. F. Meggers and which he has kindly offered to the committee. It is hoped that this important contribution can be made available to the Society membership.

Subcommittee III on Quantitative Methods and Applications (M. L. Fuller, chairman).—No further work has been carried out on the preparation of methods of sampling, following the decision by the committee last year to defer to Committee E-3 on Chemical Analysis of Metals on this matter.

Certain revisions of, and additions to two of the tentative methods for quantitative spectrochemical analysis of zinc and lead, under the jurisdiction of Committee E-2, have been formulated to meet a need for broader application than was anticipated when these methods were issued in 1935. At the meeting of Committee E-2 held during this annual meeting these revisions were discussed and approved for submission to letter ballot of the committee. The nature of the contemplated changes is indicated below. These revisions after approval by letter ballot will be submitted to the Society subsequent to the annual meeting through Committee E-10 on Standards.[1]

In connection with the Tentative Method of Test for Quantitative Spectrochemical Analysis of Zinc Alloy Die Castings for Minor Constituents and Impurities (E 27 – 35 T), a need has developed for providing an optional additional test. Section 7 of the Standard provides a means of testing for compliance with A.S.T.M. specifications on composition.

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[1] See Editorial Note p. 530.

The proposed revision provides a means of extending this test to permit an approximate estimation of the actual amounts of the several constituents and impurities of the sample. Such a determination is not always required, but it is believed by the committee that provision for it will increase the usefulness of the general method.

In the Tentative Method of Test for Quantitative Spectrochemical Analysis of High Grade Pig Lead (E 25 – 35 T), provision is made only for the determination of copper, bismuth, silver and nickel. Other impurities are often present which could be determined by spectrochemical analysis, and the proposed revision will extend the scope of the method to include such other impurities.

Subcommittee V on Standards and Pure Materials (Thomas A. Wright, chairman).—This subcommittee has prepared an extensive report on the sources and characteristics of super-purity metals suitable for use as spectrographic standards. It believes that these metals can be made available to members of the committee, if satisfactory means for handling and distribution can be developed. The accumulation of the information given in this report has involved an enormous amount of time and labor in correspondence and discussion with producers of the various metals. It is a pleasure to acknowledge the splendid spirit of cooperation that has been shown by these producers, many of whom have willingly devoted a great deal of time and research effort, not only to the study of the difficult problems of preparing metals of extreme purity, but also to the actual production of such metals. Some of them have undertaken this work specifically for the benefit of the committee and without the hope of any substantial return to themselves.

It is our belief that the information contained in the report appended hereto is of outstanding importance, not only to the members of the committee and the Society, but to spectroscopists in general.

The chairman of Subcommittee V has prepared a carefully considered discussion of the need for super-purity metals, the characteristics they must have and the precautions that must be observed during their preparation, distribution, and use. This discussion is presented in an Appendix to this report.

The committee has recommended that steps be taken to arrange, if possible, for the distribution of these pure materials and other standard samples that may be available through the National Bureau of Standards.

Respectfully submitted on behalf of the committee,

H. V. CHURCHILL,
Chairman.

C. C. NITCHIE,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting Committee E-2 on Spectrographic Analysis presented to the Society on August 26, 1937, through Committee E-10 on Standards the following recommendations:

Proposed Revisions of Tentative Methods of:

Quantitative Spectrochemical Analysis of High Grade Pig Lead for Copper, Bismuth, Silver and Nickel (E 25 – 35 T), and

Quantitative Spectrochemical Analysis of Zinc Alloy Die Castings for Minor Constituents and Impurities (E 27 – 35 T).

The above recommendations were accepted by Committee E-10 and the methods in their revised form have been issued as 1937 Supplements to the publication of "A.S.T.M. Methods of Chemical Analyses of Metals, 1936."

REPORT OF SUBCOMMITTEE V ON STANDARDS AND PURE MATERIALS

The present status of the project on super-pure metals is covered in the report for the respective metals and for the respective cooperating companies who have produced, are producing, or have signified their willingness to produce the highest possible purity metals.

Availability, unless otherwise noted, is dependent upon arrangements for a distributing agency. No orders should be addressed to the companies direct, except as indicated.

Purity ratings or figures where given are those furnished by the producer, unless otherwise noted.

Unless otherwise noted, the degree of accuracy of any figures are subject to consideration such as those mentioned in the paper on "Super-Purity Metals," by Thomas A. Wright, published as an Appendix to this report. No responsibility for the figures given therein is assumed by the committee or the Society.

ALUMINUM, SUPER-PURE

Producer: Cie. de Produits Chimiques et Electrometallurgiques, France, Mr. Gaston G. Gauthier, Chief, Research Laboratories.

Form, Cost, Amount and Availability: Not yet known. Negotiations in progress.

Prospects: Excellent.

Purity:

Aluminum (Note)	99.997 to 99.991	per cent
Iron	0.0012 to 0.0013	per cent
Silicon	0.0015 to 0.0037	per cent
Copper	0.0003 to 0.0035	per cent
Conductivity (per centage of copper)	65.45 to 65.35	per cent

NOTE.—Copied from "The Conductivity of Super-Purity Aluminum: The Influence of Small Metallic Additions" by Mr. Gaston G. Gauthier, *Journal*, Inst. Metals (British), Vol. LIX, 1936.

Negotiations are in progress with The British Aluminum Co. which, it is hoped, will make available a super-pure aluminum of possibly 99.995 with copper, silicon and iron the major impurities. This, of course, is not a commercial grade and has not been heretofore available.

ANTIMONY

Producer: Texas Mining and Smelting Co.

Form and Cost: Not yet known.

Availability: Best commercial grade "Lone Star," guaranteed and sold on a basis of minimum 99.80 per cent antimony, with arsenic not over 0.05 per cent, and any other element not over 0.10 per cent.

Purity:

	TYPICAL RANGE OF ANALYSIS (NOTE 1)	AVERAGE ANALYSIS
Antimony	99.85 to 99.90 per cent	99.88 per cent
Iron	0.015 to 0.030 per cent	0.025 per cent
Arsenic	0.015 to 0.030 per cent	0.025 per cent
Lead	0.013 to 0.020 per cent	0.018 per cent
Copper	trace to 0.010 per cent	0.006 per cent
Bismuth	None	.....
Zinc	None	.....
Sulfur	0.030 to 0.040 per cent	0.035 per cent

NOTE 1.—Analysis (presumably chemical) furnished by producer.

NOTE 2.—Producer will consider undertaking production of their highest purity grade for the benefit of the committee. Purity, analysis, form and grade have not been ascertained or agreed upon, but small sample has been received though not opened and examined.

BISMUTH, SUPER-PURE

Producer: Cerro de Pasco Copper Corp.

Form: Shot or splash sample, small 1 to 1.5-oz. bars.

Cost: Approximately $10.00 to $15.00 per pound when, as and if analyzed.

Amount: Approximately 100 lb.

Availability: At discretion of committee.

Purity: 99.9984 per cent or better.

	TYPICAL ANALYSIS OF PRESENT HIGHEST-PURITY METAL (NOTE 1)
Silver	trace to 0.0003 per cent (Note 2)
Copper	0.0001 to 0.0002 per cent
Lead	0.0006 to 0.0010 per cent (Note 2)
Antimony	0.0001 to 0.0002 per cent (Note 2)
Iron	0.0001 to 0.0004 per cent
Arsenic	nil by Betterdorf test
Tellurium	nil (see arsenic above)
Tantalum, Gallium, Indium and Cadmium	not known

NOTE 1.—Analysis (presumably chemical) furnished by producer.

NOTE 2.—Can possibly be reduced in the super-purity grade contemplated.

CADMIUM, SUPER-PURE

Producer: New Jersey Zinc Co.

Form: Crystals (nodules).

Cost: $40.00 per lb. (minimum order 2 oz.).

Amount: 10 lb.

Availability: To committee or distributing agency.

Purity: Possibly well over 99.99 per cent. Impurities—iron or copper or magnesium or lead, when present so low that no quantitative method has as yet been developed. Not all impurities mentioned are present in any one batch.

NOTE.—The Consolidated Mining and Smelting Co. of Canada is said to have produced a cadmium metal of exceptionally high purity but data is not at hand as to complete analytical study. Its purity as published is said to be at least 99.999 per cent and to be "spectroscopically pure."

COPPER

Producer: U. S. Metals Refining Co.

Form: Cut cathode or powder.

Cost: Not known as yet.

Amount: Not known as yet.

Availability: Not known as yet.
Purity: Still in experimental stage.

Note.—Adam Hilger, Ltd., supplies copper rods 5 mm. by 17.5 cm. of a rated purity of 99.994 per cent (Metals = 0.006 − oxygen trace).

Iron, Super-Pure

Producer: Westinghouse Electric and Mfg. Co. (Research Laboratory).
Form: Can be rolled to a size selected by the committee.
Cost: $5.00 per lb.; Minimum order 1 lb.; no analysis furnished.
Amount: ±200-lb. lots.
Availability: 1 bar of 200 lb. on hand, not rolled as yet.
Purity: 99.95 ± per cent.
Analysis: Data below for a lot made 2 yrs. ago. Except for the carbon and oxygen this analysis was performed by Mr. Sam. Judd Lewis, London, England, consultant for Adam Hilger, Ltd.

Silicon	less than 0.010 per cent
Oxygen	less than 0.010 per cent
Carbon	less than 0.005 per cent
Sulfur	less than 0.005 per cent
Nitrogen	less than 0.004 per cent
Phosphorus	less than 0.003 per cent
Manganese	less than 0.003 per cent
Zinc	less than 0.002 per cent
Molybdenum	less than 0.001 per cent
Copper	less than 0.001 per cent
Lead	less than 0.0004 per cent
Tin	less than 0.0002 per cent
Barium	less than 0.0001 per cent
Titanium	less than 0.0001 per cent
Silver	slight traces, but each less than 0.00005 per cent
Aluminum	
Arsenic	
Boron	
Calcium	
Cobalt	
Chromium	
Gallium	
Iridium	
Magnesium	
Nickel	
Vanadium	
Tungsten	
Germanium	not run
Antimony	not run
Iron (by difference)	more than 99.95 per cent

Note.—One of the iron carbonyls is said to have been used abroad in studies of the wave-lengths of iron.

Lead, Super-Pure

Producer: American Smelting and Refining Co. (Research Department).
Form: Small bars ±1 lb.
Cost: Not determined.
Amount: Several tons.
Availability: 100 to 200 lb. at disposal of committee.
Purity: Probably minimum of 99.9999 per cent.
Analysis: Chemical and Spectrographic.

Bismuth	less than 0.00001 per cent[a]
Copper	less than 0.00004 per cent[a]
Silver	less than 0.00001 per cent[b]
Tellurium, Arsenic, Antimony, Tin	each less than 0.00001 per cent[c]
Iron	0.0001 per cent[d]
Cadmium	less than 0.0001 per cent[e]
Zinc	less than 0.0001 per cent[e]
Nickel	less than 0.0001 per cent[e]
Cobalt	less than 0.0001 per cent[e]
Thallium	less than 0.0001 per cent[e]
Gold	less than 0.0001 per cent[e]

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[a] Determined spectrographically with special technique.
[b] Not detectable spectrographically with special technique.
[c] Not detectable spectrographically even from a precipitate from a 1000-g. sample.
[d] Can be removed by remelting and cleaning in glass.
[e] Not detectable chemically or spectrographically.

MAGNESIUM, SUPER-PURE

Producer: Dow Chemical Co.

Form: 1-lb. ingots. Possibly also as small bars ½ in. square by 6 in. long.

Cost: $2.00 per lb. with analysis in 50-lb., min., lots.

Amounts: Minimum order, 50 lb.

Availability: In 50-lb. lots to order of committee or distributor designated by them.

Purity: Not less than 99.97 per cent.

Analysis: Typical as furnished by producer by combined chemical and spectrographic methods:

Sodium	0.005 per cent
Aluminum	less than 0.005 per cent
Iron	less than 0.001 per cent
Silicon	less than 0.001 per cent
Nickel	less than 0.0002 per cent
Manganese	less than 0.0002 per cent
Copper	less than 0.0001 per cent
Gases (H_2, N_2, etc.)	a few thousandths of one per cent
Magnesium (by difference)	more than 99.97 per cent

With the exception possibly of potassium (the concentration of which is possibly similar to sodium) no other metals are detectable by ordinary methods of spectroscopic analysis.

NICKEL

Producer: International Nickel Co.

Form: Rectangular pencils sheared from a cathodic deposit.

Cost: Not determined definitely.

Amount: ±1000 g. expected.

Availability: Present plans to make up free for the committee do not include provision for packing and distributing or for cost of analysis.

Purity: As high or higher than any yet experimentally produced. Definite data will require development of special painstaking analytical methods.

Analysis: Analyst to be arranged for or possibly recommended by the committee.

NOTE.—Adam Hilger, Ltd., supplies nickel in flakes of a rated purity of 99.97 per cent (iron 0.004 to 0.01 per cent, carbon and oxygen, traces).

TIN, SUPER-PURE

Producer: Vulcan Detinning Co.

Form: Small bars ±95 g. as melted in heat-resistant glass and cast in porcelain.

Cost: Not as yet determined.

Amount: 500 g.

Availability: Pending arrangements for distribution and analysis.

Purity: (99.995 per cent ± 0.002 per cent?) Not as yet precisely determined, but probably as high or higher than any other available.

Analysis: Arrangements for analysis still to be made. A preliminary and partial spectrographic analysis contributed by Lucius Pitkin, Inc., indicates an impurity content of the following order:

Lead	not detected
Copper	less than 0.0001 per cent
Iron	±0.0005 per cent (as first washed in HCl)
Silver	±0.0001 per cent (Note)
Magnesium	±0.0002 per cent (Note)
Arsenic and Antimony	possibly of the order of 0.004 per cent (Note)

NOTE.—High-purity carbon anodes are perhaps the source of the silver and magnesium, and some arsenic may have come from the glass in which melted.

The American Smelting and Refining Co. has made, for its own use, an extraordinarily high-purity tin, but the amount was small and it is no longer available.

ZINC, TWO GRADES

S. P. Grade

Producer: New Jersey Zinc Co.

Form: Crystals (as distilled).

Cost: $20.00 per lb.

Availability: Not for general distribution.

Purity: 99.9999 per cent.

Analysis: Lead line suspected, no other impurities by conventional spectrographic analysis.

C. P. Grade

Producer: New Jersey Zinc Co.

Form: Sticks $\frac{1}{4}$ in. in diameter by 8 in. in length, weight approximately 45 g.

Cost:

2 sticks ±90 g. or ±3 oz.—minimum order	$3.00
5 sticks ±225 g. or ±$\frac{1}{2}$ lb	6.00
10 sticks ±450 g. or ±1 lb	10.00
2 lb	16.00
5 lb	30.00
10 lb	50.00

Availability: May be procured from Lucius Pitkin, Inc., 47 Fulton St., New York City.

Amount: 200 lb.

Purity: 99.998 per cent, by difference.

Analysis: By Lucius Pitkin, Inc. Spectrochemical analysis for lead, copper, iron, cadmium and tin. Chemical analysis for arsenic.

Lead	0.00015 per cent
Copper	0.00003 per cent
Iron	0.00100 per cent
Cadmium	0.00005 per cent
Tin	less than 0.00005 per cent
Arsenic	less than 0.00001 per cent
Zinc (by difference)	99.9987 per cent

SILVER

Producer: Handy & Harman.

Arrangements pending. Probably ready within a year. Adam Hilger, Ltd., supplies cast rods of a rated purity of 99.99 per cent containing but the most minute trace (?) of Copper.

Handy & Harman have produced ⅜-in. rod for use as spectroscopic electrodes which, by present analytical technique, has a purity of 99.993 per cent, or perhaps better.

GOLD

Producer: Previous high purity made by Lucius Pitkin, Inc. None available. Spectrographic evidence showed faint copper line by arc but not by spark. No lead line discernible. A "proof gold" grade is available in thin rolled strips, but demand has not justified any complete analysis. Purity ordinarily is over 99.998 per cent.

Proof gold made by the U. S. Mint or Assay Offices, for their own use only of course, is thought at times to reach a purity of 99.998 per cent also.

PLATINUM

A very high purity wire, carefully tested for maximum conductivity and minimum resistivity, has been made and is available from Sigmund Cohn, 44 Gold St., New York City at a price which naturally carries a premium above the market price of commercial platinum.

They estimate the purity to be possibly as high as 99.9999 per cent based primarily on electromotive force and, to a more restricted extent, on spectrographic measurements.

Baker & Co. have produced high-purity platinum and palladium which by conventional methods have a purity higher than 99.999 per cent and will on order engage to produce as high-purity metals as can be made within permissible costs.

Adam Hilger, Ltd., supplies platinum in 3-mm. rod of a rated purity of 99.999 per cent.

MANGANESE

Not yet undertaken for committee pending extent of demand. No negotiations in progress.

CHROMIUM

Same as for manganese.

TITANIUM

Same as for manganese.

MOLYBDENUM

Same as for manganese.

CARBON

There appears to be a field for super-purity carbons (Gereinigte Spektralkohlen) (of the carbon, not graphitized carbon, type) such as are available abroad from Gebr. Ruhstrat A. G., Goettingen.

MISCELLANEOUS

Certain metals and salts designed for use as spectrographic standards may also be procured from Adam Hilger, Ltd., or through their agents in this country. Salts or compounds may at times be preferable even to the pure metal and will be considered by the committee.

SUMMARY

This report is offered with the understanding that the companies mentioned are not to be approached direct by readers unless specifically so noted.

The report is further designed to encourage a flow of information to the committee on production, methods of checking and retaining purity, and of availability, forms, and cost.

Respectfully submitted on behalf of the subcommittee,

THOMAS A. WRIGHT,
Chairman.

APPENDIX

SUPER-PURITY METALS

By Thomas A. Wright[1]

The subject of high or super-purity metals is more complex than may appear at first sight. To procure and make available such metal requires consideration of at least the major factors to be enumerated. Obviously, the production is best carried out by an organization aware of the desirability of having available such unusual material, staffed by an interested and capable scientific personnel, equipped with proper facilities or prepared to install them and not unmindful that in large part they are making a contribution to their fellow workers in science and industry. Rarely, in the initial stages at least, will there be any monetary return commensurate with the task or the responsibilities involved. The reward is rather in the task itself and in the gratitude of one's fellows and the resultant contributions to science that will ensue. Later may come tangible returns.

In this project of Subcommittee V on Standards and Pure Materials, of the Society's Committee E-2 on Spectrographic Analysis, the purpose is primarily to make available metals which may serve as basic, fundamental spectrographic standards. Science progresses by cooperation, however, so sight has not been lost of the fact that such super-purity metals, once available, will, in the next decade, be in great demand by other chemists, research metallurgists and physicists. It is well that this is so, for greater demand will permit of attaining more collateral checks as to purity, greater improvement in production and handling and, incidentally, lower unit costs. Fortunately for the spectroscopist, high as the premium in some instances may appear, the actual cost due to the relatively small amounts used will be relatively small compared to that which a research metallurgist would encur.

If one were to describe the ideal super-purity metal, it should meet requirements such as the following:

1. Highest purity possible within our present technique,
2. Be accompanied by a report of analysis,
3. Be obtainable in various forms,
4. Be obtainable in quantities from 1 g. up,
5. Storage and container conditions should be carefully controlled,
6. Ease of recontamination should be recognized,
7. Distribution should preferably be from a centralized point,
8. Cost should not be prohibitive, and
9. All of the several metals used regularly as the base or major components of alloys at least should be available.

These factors warrant more detailed study.

Purity:

The ideal or truly pure metal is, of course, the element as such for we have come to a greater realization in the last decade that some of our really high-purity metals, for certain purposes and from an increasingly wider viewpoint, are, in effect, alloys. We have revised certain of our physical constants as more refined metals have become available and we may change some of our concepts. Nevertheless, it seems necessary at this time to eliminate from our major consideration, at least, the subject of gases in these metals. And we shall use the term "super-purity" (or S.P.) rather than the misnomer "spectroscopically pure," or the even more indefinite and unsatisfactory term "chemically pure." Both of the latter terms are far too dependent upon the technique employed in analysis to be justified. Extraordinarily pure as one or two available metals are, the author knows of none to which the term "spectroscopically pure" may be scientifically applied though two at least will defy proof that they are not what we might call "chemically pure," if we could call anything such. For the time being such terms, when so employed by the producer, will be used in order to identify them.

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[1] Technical Director and Secretary, Lucius Pitkin, Inc., New York City.

Several other factors come to mind. One is the concentration of what we must here call "impurities," that is, inherent or attendant elements; and the other is contaminants. In considering these we shall also have most in mind at present those attendant elements which disclose themselves, or should, with proper emission spectrography technique, even though that may require either a specific emission procedure or a prior chemical concentration.

In refining a metal it is not, as might be first thought, always necessary to start with one of the commercially refined grades. On the contrary, quite the reverse may be true, particularly if there is some major impurity which can be precipitated and thus co-precipitate and thereby eliminate all or a greater part of other allied impurities. This may be of particular advantage if there is some one or more elements present which otherwise may be concentrated from stage to stage, for example, of resublimation or electro-deposition. One must also keep in mind that there are other than the conventional impurity elements which we may not usually think of as being present. These, too, must be removed but may require the development of a special process or a combination of purification and refining procedure.

Again in the refining, whether it be electrochemical or what, a major difficulty may be the introduction of similar or foreign impurities. Prevention of contamination, particularly if melting has been or must be resorted to, may, as a matter of fact, be the most difficult phase of production, and for some metals an almost unsurmountable one. So much so that for certain purposes, requiring a freedom from contamination which is in some cases not attainable by any one melting procedure, it is conceivably possible that one type of crucible may be indicated in one case and a second type in another. Sodium, so common in perspiration, in ceramics, and in the atmosphere along the coast offers, in its avoidance, particular difficulty. Atmospheric dust is to be avoided.

As a further illustration of the extraordinary care needed, tin may serve as an example as melted in a quartz or a heat-resistant glass and cast in small bars, using a porcelain combustion boat as a mold. Yet one must consider and check whether certain constituents of the glass may not have recontaminated the melt. It is true that one may prepare this metal conveniently by pouring into distilled water to make a shot or moss and for some purposes this might be more desirable than small bars. Fine sawings or raspings, however, are seemingly very convenient for a spectroscopist where a solid sample is being arced. But, when so prepared, it is imperative that the sawings be washed in acid to remove the adherent film of iron introduced from use of a hack saw blade or rasp. Experience has shown that despite the care taken in selecting a clean smooth blade, as much as 0.003 per cent of iron may thus be introduced into tin sawings.

Analysis:

Even in the production of as high as 100 or 200 lb. of a super-purity grade of metal, expensive and time consuming as that may be, the cost may be less than that of obtaining a correct conception of the actual purity. The analysis of such metals is no mean problem requiring, as it may, a carefully planned prior chemical separation, isolation or concentration of the suspected impurities. At first glance it might be thought that the metal would serve as a standard provided that the regular spectrographic technique employed would not disclose the presence of "x" element. (No doubt this is true in many cases for there is never any warrant for using a needlessly pure but expensive metal. One must, however, be prepared to define the word "needlessly.") More considered thought will show this is not the case. For example, one may be employing two of these metals to make a standard alloy, only one of which has shown either arsenic or cadmium in amounts too small for either to be resolved by the spectrograph available when examined for by conventional spectrographic methods, and while we may not report them as being in the other metal "absent" (a term unfortunately used erroneously too often) yet we would report (for routine or normal purposes) "not detected" or "not found." In the type of studies contemplated under this project, if those terms are employed, we must limit them by defining the technique employed.

We must also recall that spectrography as applied to metallurgy is a means, not an end in itself. It is an art, or science, if you will, employed as one phase of a research, development, or exploratory study or for control of purchases and production. It may be essential in the first three items that no element goes undisclosed for we know that metallurgical effects may be additive, that some of our so-called facts are conceptions only and some may be misconceptions, that the presence of one element may offset another, that all attendant elements are not necessarily impurities, though we usually think of, and call them such, and that we have a growing list of examples of the importance of one-time "trace" elements. If the chemist or metallurgist may not ignore the fourth or sixth decimal place, what shall one say of the physicist in his determination of electrical resistivity or of the student of corrosion tracking down fundamentals.

We have then the problem of concerted attack on the methods of analysis to be employed, which for others than the spectroscopist must sooner or later involve the determination of the gases and other elements not within the province of conventional arc or spark emission spectrography. Fortunately, a start has been made and certain of the metals now or to be made available will be furnished with at least partial analyses. Wemco iron, S.P. zinc and the very new 99.9999+ per cent lead are particularly good examples of the very difficult type of analytical technique required, the data being supplied in this case from the laboratories of the producers. Conventional methods carried out by unconventional chemists must be supplemented by all the tools, ingenuity and skill available. Spectrography, macrochemical analysis, chemical microscopy, microchemistry, spot tests, fluorescence, all come to mind. It may even be that end and by-products must be examined as a check in some cases on efficiency of elimination. Certainly those who will tackle this analytical nut must be of an inquiring nature and capable of self criticism.

Form:

The form in which such materials are available depends primarily on the purification procedure. Cementation, sublimation or even electrodeposition may give a powder where the spectroscopist may prefer a bar or rod suitable as a self-electrode or capable of being drawn into wire. An unusually high-purity gold made some years ago for the Cancer Research Institute (Crocker Foundation) serves as an illustration. Yet a cathode may also permit of cutting into strips though pickling may needs follow to remove tool contamination. Shot and moss have been mentioned. Powder and rod of $\frac{3}{16}$ in. in diameter appear to be often the more desirable but rod may be compressed from powder.

Weights:

Requests as to amounts desired range in weight from a single gram to as much as 3 kg. Quantities of from 1 oz. to 1 lb. will cover most demands of spectroscopists. For some studies of a fundamentally precise nature 1 lb. of some metals may be little enough.

Storage:

If super-purity metals are produced they must be kept so. Dust, moisture and atmospheric impurities in general must be avoided. Transparent wrappings sometimes employed for high-purity carbons and metal rods must be examined of themselves and selected carefully for freedom from inherent or coating contaminants. An acetate wrapper may be preferable even to a moisture-proof cellophane. In any event, an acid wash is advisable just prior to use, where no harm and much good may ensue, particularly if the material has been handled. One may not, for example, handle super-pure aluminum with the fingers without assuming that sodium has contaminated it. Glass containers should be wear-resistant and preferably fitted with a screw closure of the plastic type containing no metal insert. Glass-stoppered bottles should be avoided as offering ready ingress of settled dust. If the metal has been made into standard solutions, initial calibration at least may require duplicate solutions made up in quartz or in some solution-resistant glass. Arsenic, zinc and boron may be introduced from a glass container, and, of course, sodium, silica, and calcium.

Recontamination:

As will have been noticed the matter of recontamination has cropped up here and there throughout this paper. This is as it should be. The possibility should never be lost sight of.

Distribution:

Distribution is a problem. Organizations dealing in carload lots but which will readily agree to produce or which have produced super-pure metals naturally do not wish to be bothered with parcelling out small items like a pound, much less an ounce or a gram. Nor do they lightly engage some other organization unless they feel certain that the attendant grave responsibilities are appreciated and that the high purity so laboriously attained will be respected and retained. While the author's organization is distributing one high-grade metal—not the very highest, however—and is even furnishing with each order a copy of the analysis covering the six important impurities and may shortly handle one or two more other metals, but of the super-purity type, nevertheless, there is a growing demand for a central distributing agency. This is quite understandable.

The National Bureau of Standards has been suggested for obvious reasons. There is little that can be said against the plan and much to commend it. It is possible that one or more producers or sponsors may agree to supply a certain amount free in the event the Bureau would engage to make the required analysis or check analysis. Incidentally, it is also possible that some S.P. metals may be made available without cost to members of Committee E-2, but *without analyses* on condition that they report their findings.

Cost:

The price will vary over a wide range. It may cost as much to produce a gram of one metal as a pound of another. The cost of the analysis may be far greater than the cost of production and must always be spread over the weight produced whether it be 500 g. or a ton. Overhead handling charges are as great for 1 g. as for 100 lb. The cost of the container and of transportation may vary but that is all, except in some cases it would be less costly to give away an ounce or so than to weigh out a gram.

The Metals:

At present some super-purity metals have been produced but are not available, some are in the experimental stages, some have been produced but are not analyzed, some are yet to be started in production and some are produced, in part analyzed, and it only remains to arrange for distribution, and one or two are available. All except one are of American production. Effort has been concentrated on the following twelve elements, the assumed composition being:

Aluminum	99.997 per cent	Lead	99.9998+ per cent
Antimony	99.90+ per cent	Magnesium	99.97+ per cent
Bismuth	99.998+ per cent	Nickel	?
Cadmium	99.99+ per cent	Silver	±99.999 per cent
Copper	?	Tin	99.99+ per cent
Iron	99.995+ per cent	Zinc	99.999+ per cent 99.998+ per cent

Gold and platinum have both been produced in a super-pure state. The latter may be available in wire form checked for purity by extended work on resistivity.

Carbon, while not a metal, is of paramount importance to the spectroscopist and is a source of continued trial and investigation, particularly as to graphitized rod form. Ungraphitized carbon for electrodes appears to be foreign in production and preferred abroad.

Availability:

Until such time as proper arrangements have been made for distribution it does not seem wise to publish the addresses of sources.

REPORT OF COMMITTEE E-3

ON

CHEMICAL ANALYSIS OF METALS

Committee E-3 on Chemical Analysis of Metals held one meeting during the year, on June 30, 1936, at Atlantic City, N. J., during the annual meeting of the Society. By-laws governing the activities of the committee, supplementary to the general Society's Regulations, have been prepared and approved by letter ballot of the committee. On the basis of these by-laws a more complete subcommittee organization has been developed.

At a meeting of the committee held on June 30, 1935, at New York City, the following resolution was considered at length and was approved in substance and referred to the Advisory Committee for further study before submission to letter ballot of the committee:

> Methods of sampling and analysis devised by Committee E-3 on Chemical Analysis of Metals shall show as close an approach to accuracy as is in accord with allowable costs and good analytical practice.

During the year 24 new members have been added and 1 member resigned, leaving a net increase of 23 members and bringing the total membership of the committee up to 50.

Ferrous Division.—Subsequent to the last annual meeting of the Society, the Ferrous Division completed the revision of analytical methods for the determination of some 17 elements in steel and iron, which were presented[1] to the Society through Committee E-10 on Standards. These methods, entitled Tentative Methods of Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron and Wrought Iron (E 30 – 36 T),[2] were accepted for publication as tentative on August 26, 1936, to replace the following standards which were accordingly withdrawn:

Standard Methods of:

- Chemical Analysis of Plain Carbon Steel (A 33 – 24),
- Chemical Analysis of Alloy Steels (A 55 – 24),
- Sampling Rolled and Forged Steel Products for Check Analysis (A 130 – 30), and
- Sampling and Chemical Analysis of Pig and Cast Iron (A 64 – 27).

The committee also recommended[1] through Committee E-10 that all of the existing standard methods of chemical analysis under its jurisdiction applicable to the ferro-alloys and the non-ferrous metals, as indicated below, be reverted to a tentative status since it planned further to develop and improve the analytical procedures on these subjects and to consolidate certain of the present methods:

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee E-3 reported results of the letter ballot vote as follows: Of a total membership of 28, 25 members returned their ballots, all of whom voted affirmatively.

[2] A.S.T.M. Methods of Chemical Analyses of Metals, p. 1, 1936.

Standard Methods of:

Sampling Ferro-Alloys (A 103 - 27),
Chemical Analysis of Ferro-Alloys (A 104 - 27),
Chemical Analysis of Ferrotungsten and Ferromolybdenum (A 104 - 34),
Chemical Analysis of Alloys of Lead, Tin, Antimony and Copper (B 18 - 21),
Chemical Analysis of Manganese Bronze (B 27 - 19),
Chemical Analysis of Gun Metal (B 28 - 19),
Battery Assay of Copper (B 34 - 20),
Chemical Analysis of Pig Lead (B 35 - 24),
Chemical Analysis of Slab Zinc (Spelter) (B 38 - 21),
Chemical Analysis of Aluminum and Light Aluminum Alloys (B 40 - 35),
Chemical Analysis of Nickel (B 41 - 23),
Chemical Analysis of Brass Ingots and Sand Castings (B 45 - 27),
Chemical Analysis of Bronze Bearing Metal (B 46 - 27), and
Chemical Analysis of Silver Solders (B 81 - 33).

This recommendation was accepted and all of the above standards were reverted to tentative and are published in "A.S.T.M. Methods of Chemical Analyses of Metals, 1936."

The Ferrous Division, after a brief respite from its activities, is studying methods for the determination of aluminum, columbium, boron and nitrogen, and has undertaken a revision of the Tentative Methods of Chemical Analysis of Ferro-Alloys (A 104 - 36 T).

Non-Ferrous Division.—The first meeting of this division was held in Washington, D. C., on November 13. A general discussion was held on the problem of revising the present A.S.T.M. methods for the analysis of non-ferrous alloys. It was decided to establish a number of subcommittees. The respective chairmen were appointed later by the division chairman, as follows:

I on Copper, Nickel and Their Alloys (R. P. Nevers, chairman),
II on Lead, Tin, Antimony, Bismuth and Their Alloys (J. B. Mosley, chairman),
III on Aluminum, Magnesium and Their Alloys (J. G. G. Frost, chairman),
IV on Zinc and Its Alloys (R. W. Coltman, chairman), and
V on Precious Metals and Their Alloys (R. H. Leach, chairman).

Subcommittees I, II and IV are engaged, respectively, in the revision of the Tentative Methods of Chemical Analyses of Manganese Bronze (B 27 - 36 T), of Alloys of Lead, Tin, Antimony and Copper (B 18 - 36 T), and of Slab Zinc (Spelter) (B 38 - 36 T).

There has recently been referred to the Non-Ferrous Division a request from Committee A-5 on Corrosion of Iron and Steel for the development of a procedure for the determination of small quantities of aluminum in zinc.

At the request of Committee B-7 on Light Metals and Alloys, Cast and Wrought, it also plans to prepare procedures for the analysis of aluminum-base alloys for both permanent mold and sand castings.

Sampling Division.—This Division will be organized shortly and will undertake a study of the sampling provisions appearing in the various tentative methods of chemical analysis of non-ferrous metals and alloys.

This report has been submitted to letter ballot of the committee which consists of 38 members; 30 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. E. F. LUNDELL,
Chairman.

H. A. BRIGHT,
Secretary.

REPORT OF COMMITTEE E-4

ON

METALLOGRAPHY

A meeting of Committee E-4 on Metallography was held during the annual meeting of the Society in Atlantic City, N. J., on July 2, 1936. The important activities of the committee during the year have been the work of Subcommittee VI on X-ray Methods in preparing for publication the Symposium on Radiography and X-ray Diffraction Methods,[1] which was held at the 1936 annual meeting, and the organization of a Special Subcommittee on Grain Characteristics of Steel for the purpose of revising the Standard Grain Size Chart for Classification of Steels (E 19 – 33).

Committee E-4 records with deep regret the loss of one of its valued members in the death on December 16, 1936, of Dr. William Campbell.

Activities of Subcommittees

Subcommittee IV on Photography (L. V. Foster, chairman).—The Standard Rules Governing the Preparation of Micrographs of Metals and Alloys, Including Recommended Practice for Photography as Applied to Metallography (E 2 – 36), revised last year, were submitted in their amended form to the American Standards Association for approval as American standard. The rules were approved on October 21, 1936, and have been assigned the A.S.A. designation: Z30.1–1936.

Subcommittee VI on X-ray Methods (H. H. Lester, chairman).—With the resignation of R. F. Mehl, the chairmanship of this subcommittee has been placed in the hands of H. H. Lester of the U. S. War Department, Watertown Arsenal. The subcommittee has been actively engaged during the year in connection with the publication of the Symposium on Radiography and X-ray Diffraction Methods[1] which has been issued as a separate volume of 340 pages. This compilation constitutes a valuable addition to the literature, and fills a very definite need as is evidenced by the demand for copies.

Subcommittee VI held a meeting in New York City on February 17, 1937, to discuss the scope of its activities. As a result, the following proposals were recommended:

1. That a separate standing committee of the Society be organized to consider the subject of radiography;

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[1] Published by the American Society for Testing Materials (1936). (Available as separate publication.)

2. That the study of X-ray diffraction methods be continued as the principal activity of Subcommittee VI; and
3. That the chairman of Committee E-4 be requested to invite various metallurgical societies to be represented at a meeting for the purpose of discussing the formation of a general committee on X-ray diffraction work in industry.

These proposals were submitted to letter ballot of the subcommittee, which consists of 10 members; 7 members returned their ballots, all of whom voted affirmatively.

The first two proposals will receive the attention of Committee E-4 at its next meeting, to be held during the approaching annual meeting of the Society.

In line with the third proposal, the Chairman of Committee E-4 arranged a joint meeting on April 15, in New York City, of representatives of the American Institute of Mining and Metallurgical Engineers, American Society for Metals, American Institute of Physics and the A.S.T.M., at which it was decided that useful work could be accomplished by holding informal conferences under the sponsorship of the American Institute of Physics for a discussion of the fundamental problems of metallurgy. Among other things, these conferences would point out the best applications of X-ray diffraction methods. The first of these conferences is to be held on June 28, during the annual meeting of the Society in New York City, and will be devoted to considerations of the physical basis of metal properties.

Special Subcommittee on Grain Characteristics of Steel (M. A. Grossmann, chairman).—This special subcommittee has been reorganized under the chairmanship of M. A. Grossmann of the Carnegie-Illinois Steel Co. The membership is as follows:

M. A. Grossmann (chairman)
R. H. Aborn
O. E. Harder
H. B. Knowlton
H. W. McQuaid
Reinhold Schempp
E. E. Thum
J. F. Wyzalek

A meeting of the subcommittee was held on March 3 in Chicago, during the spring group meetings of A.S.T.M. committees, for the purpose of considering a proposed grain size standard which meets some of the objections to the present Standard Grain Size Chart for Classification of Steels (E 19 – 33). The proposed chart has been circulated in the subcommittee and will be reported at the next meeting of Committee E-4.

This report has been submitted to letter ballot of the committee, which consists of 49 members; 36 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

JOHN T. NORTON,
Chairman.

J. J. BOWMAN,
Secretary.

REPORT OF COMMITTEE E-9
ON
RESEARCH

Committee E-9 on Research is charged with the duty of annually reviewing "the progress of the Society's research activities." This is very adequately carried out through the medium of the October issue of the *ASTM Bulletin*. But this committee has also followed the procedure common to that of all active committees of the Society of rendering a report at the annual meeting. In so far, therefore, as this report covers the activities of the research committees, it is in the nature of a brief reiteration of the findings of those committees as presented concurrently at this meeting. In order, however, that those who are interested in what has been accomplished in research may have this information readily at hand in a condensed form, there is abstracted herewith some of the accomplishments in research during the year of both the research committees and the standing committees.

Research Committee Activities:

The Research Committee on Yield Point of Structural Steel has been functioning since 1926. To date it has presented five series of reports which appear in the Society's *Proceedings*, Vols. 27 to 31, inclusive.[1] A lull then occurred in its activities, but once again at this meeting it is reporting on the effect of aging on the yield point of structural steel as well as presenting its final conclusions and recommendations.[2] This late work has been possible through the cooperation of the Carnegie-Illinois Steel Co. and the Bethlehem Steel Co. in furnishing the needed steel, and the cooperation of the Rensselaer Polytechnic Institute and the University of Wisconsin, as well as the two steel companies and Lehigh University, in carrying out the tests.

This last phase of the Yield Point Committee's work has shown that there is a definite measurable effect of aging in so far as it concerns yield point and ductility. This is of such moment as to lead the committee to suggest that the metallurgist in particular should concern himself in determining the reason for this as well as to determine more adequately the magnitude of the age effects. It further recommends that the factors affecting the yield point of the various alloy steels entering the structural field be studied. Apparently, therefore, the committee indirectly recommends that its activities be continued notwithstanding its final report. The committee also makes definite recommendations as to the testing machine speeds to be used in the vicinity of the yield point and the location of the test specimens to be cut from steel shapes.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I, p. 646 (1927); Vol. 28, Part I, p. 105 (1928); Vol. 29, Part I, p. 101 (1929) Vol. 30, Part I, p. 133 (1930); and Vol. 31, Part I, p. 136 (1931).

[2] See p. 87.

The Research Committee on Fatigue of Metals reports[1] that none of its work has advanced sufficiently to permit of its publishing data or conclusions this year. However, it has been following the investigations being carried on of interest to it. It presents as information two types of nomenclature, but does not indicate now which is to be preferred.

The Joint Research Committee on Effect of Temperature on the Properties of Metals of the American Society of Mechanical Engineers and the A.S.T.M. has been successful in having pledged $19,500 for the carrying out of its work. These funds permitted enlargement of the work, and to do so effectively without undue burden on the membership, the joint committee was reorganized. Its current report[2] indicates the nature of the reorganization, the various problems now being studied, and their general status. Two of the problems are in such a stage as to warrant some detailed discussion. These are being presented at this meeting and cover "Long-Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel, and 0.35 per cent Carbon Steel" and "Discrepancies in the Load-Carrying Abilities of Carbon Steel at 850 F."

The committee is recommending the continuation as tentative of the two methods of testing metals at high temperatures, namely, the Tentative Methods of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 34 T)[3] and for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 – 35 T).[4] Revisions in the former, dealing with short-time testing are proposed, including a new method of preparation of cast tension specimens of heat-resistant metals for tests up to 2200 F.

The committee also cooperated with the Committee on Plasticity, Division of Applied Mechanics of the American Society of Mechanical Engineers, in arranging a technical session at the December, 1936, annual meeting of the A.S.M.E. At this session seven papers and reports were presented on creep and relaxation of steels.

The Joint Committee on Exposure Tests of Plating on the Non-Ferrous Metals is a group of the American Electro-Platers' Society, the National Bureau of Standards, and this Society. It reported its activities in last year's *Proceedings*,[1] giving the program of its investigations and indicating that the present work is an expansion of work previously carried out by the Electro-Platers' Society and the Bureau of Standards on exposure tests of electro-plated ferrous metals. Both the fields of ferrous and non-ferrous metals are now covered with exposures at Key West, Fla., New York City, Sandy Hook, N. J., Pittsburgh and State College, Pa., and Washington, D. C.

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[1] See p. 157.

[2] See p. 164.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 1214 (1934); also 1936 Book of A.S.T.M. Tentative Standards, p. 1307.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1291 (1935); also 1936 Book of A.S.T.M. Tentative Standards, p. 1316.

In the April issue of the Society's *Bulletin* the chairman of the joint committee presented a report covering four inspections at Key West and three at each of the other stations during the last year. No conclusions were presented, but the report clearly showed that the committee is very active and has already obtained valuable data. The results also indicate the need of further extension of the work to cover the effects of cleaning and greasing the surfaces of the coatings and the plating of further specimens using different methods of preparation.

The Joint Research Committee on Boiler Feedwater Studies is engaged in three specific studies.[2] The first of these, and the one which is receiving the most widespread attention, is concerned with the problem of caustic embrittlement. As a result of certain disclosures growing out of the committee's studies that have indicated intercrystalline cracking of boiler metal may be influenced by many more substances than sodium hydroxide, the committee is at present engaged in an effort to clarify the whole picture relating to caustic embrittlement. The studies of the committee have been divided into two distinct phases: first, investigation of chemical and physical factors that produce cracking; and, second, development and investigation of compounds that may prevent cracking. The committee has raised funds in support of this important project and now has on hand nearly $15,000 for this purpose.

The second study has to do with the method for determination of dissolved oxygen on which a final report is in course of preparation. Further work of the committee in examination of existing methods and the development of new methods, where necessary, for the determination of ions of significance in boiler feedwater and boiler water treatment has been temporarily held up pending procurement of funds to support this work.

The third project, which has only recently been initiated, deals with the application of the processes of coagulation and sedimentation in the treatment of feedwater. The immediate work has to do with the problem of removal of silica from the feedwater.

A fourth project of interest to the committee and to the industries concerned with the generation of steam is that of corrosion. While no active steps have been taken as yet in inaugurating a study of this subject, the committee has been taking an interest in various research projects in this field, including one at Purdue University in which the steam-metal reactions at high temperatures and pressures are being studied.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 212 (1936).

[2] See Semi-Annual Report of the Joint Research Committee on Boiler Feedwater Studies, ASTM Bulletin, August, 1937.

Research Committees of Other Societies on Which the A.S.T.M. Has Representation:

There are a number of organizations conducting research in which the Society is represented. The representatives make periodic reports to the Society for the benefit of all interested in the particular problem being studied. Among such are the following:

Highway Research Board, National Research Council.—Prévost Hubbard, the Society's representative on this Board, reports five major problems under study in which the membership of the Society will find much of interest. These cover the following subjects:

Effect of Freezing and Thawing on Concrete Containing Cement of Different Compositions,
Correlation of Research in Mineral Aggregates,
Volume Changes in Concrete,
Fillers and Cushion Courses for Brick and Block Pavements, and
Characteristics of Asphalts.

American Bureau of Welding.—This organization has been succeeded by a Welding Research Committee under the auspices of the Engineering Foundation, the committee being a joint effort of the American Welding Society and the American Institute of Electrical Engineers. It is now functioning with three subcommittees, on one of which—industrial research—this Society is represented by N. L. Mochel. It is suggested that the metals committees of the Society having any problems affecting welding bring them directly to Mr. Mochel's attention.

Research on Mechanical Springs.—C. T. Edgerton, the Society's representative on this research committee of the American Society of Mechanical Engineers, reports that a compendium of the present knowledge of spring materials under the title "Résumé of Spring Materials" is being prepared for publication. Fatigue tests on heavy helical springs are being continued and are expected to yield important and valuable data; there are indications that these data may require revision of methods of helical spring design and possibly changes in existing A.S.T.M. specifications.

Research Progress of A.S.T.M. Committees:

Following the usual procedure the annual review of A.S.T.M. research projects on properties of materials and methods of testing will be given in the October issue of the *Bulletin.* It is hoped that the membership will take enough time to note at least the various topics being studied. Modern conditions demand that most of us concentrate our attention on one or a very few particular fields. We become so engrossed in these that we are likely not to realize what is being done in the many fields of research in which the Society is interested. Last year's review covered almost 130 items and this year's review will present at least as many. Such broad and intensive investigations surely must convince the public that before standards have been adopted by this Society there have been thorough studies of the deportment of the materials under various conditions of usage and of test methods that will indicate quality and adequate performance.

New Activities:

The corrosion of metals is receiving the active attention of a number of committees, that is, Committees A-5, A-10, B-3, B-6, and the Joint Committee on Electro-Plating. There is also a considerable number of similar investigations being carried out abroad. As a result there has been a discussion of the advantages and needs of international coordination in this field. It has been further suggested that this Society might act as a clearing house in this country, including some arrangement that would make it possible for investigators throughout the country to keep themselves informed of each other's programs.

Committee B-6 on Die-Cast Metals and Alloys is planning to extend its researches to include exposure tests of three magnesium-base alloys.

Committee E-4 on Metallography is conferring with representative groups of the American Institute of Physics, the American Institute of Mining and Metallurgical Engineers, and the American Society for Metals to bring about discussions of the application of X-ray diffraction methods to the study of the properties of metals. A round-table discussion on this general subject is being held during the present annual meeting.

Committee D-1 on Paint, Varnish, Lacquer, and Related Products is studying ways and means of developing research on the testing of paints, with particular reference to the relationship between accelerated laboratory tests and service tests. A session of this annual meeting is devoted to a special symposium on this subject, sponsored by Committee D-1.

The need of research in the field of asphalts has been stressed recently not only in this Society but in others interested in this class of material. Through the instrumentality of J. S. Miller, Jr., one of the members of the Research Committee, two sessions are being held at this meeting at which asphalt will furnish the topic for the papers. Not only will this serve to fulfill the functions of the Society in promoting knowledge of engineering materials, but will doubtless indicate many topics for research.

Administrative Matters:

T. S. Fuller was appointed to membership on the committee to succeed Dr. H. W. Gillett, whose term expired a year ago. With this meeting the term of H. E. Smith expires and C. D. Holley has been appointed to succeed him.

Again, it is a pleasure to announce an unusually large increase in the principal of the A.S.T.M. research funds. Slightly over $373 was contributed from the Joint Committee on Investigation of Effect of Phosphorus and Sulfur in Steel, that amount remaining after the committee had completed its work. There was also added $2500 from the Society's general funds, this representing in effect part of the entrance fees which together with certain other moneys have been held in a contingency reserve. It is also a very great pleasure to call attention to the handsome contribution of about $6500 to the principal of the Research Fund from Dr. Frank O. Clements, Past-President of the Society, announced in the *Bulletin* for March. This brings the total of the principal to slightly over $19,500.

During the year the committee approved grants to various committees totaling $1450, as follows:

(*a*) $600 for the continuation of the program at the University of Illinois on the study of effect of speed of testing on the properties of concrete. This year's work is devoted to a study of inelastic deformation as affected by rate of loading and particularly by the time interval that the load is held constant at each interval of loading. The results should be of considerable value in showing the relation between plastic flow at very short periods of time and creep or flow over long-time intervals.

(*b*) $250 for the construction of a power factor test cell for Committee D-9 on Electrical Insulating Materials.

(*c*) $250 for an investigation by Committee C-10 on Hollow Masonry Building Units of methods of capping structural clay tile. This investigation will be conducted at Rensselaer Polytechnic Institute.

(*d*) $150 to Committee D-2 on Petroleum Products and Lubricants for use in revising the Viscosity Temperature Chart which constitutes one of the Society's standards.

(*e*) $200 to Committee A-3 on Cast Iron for machining test bars for fatigue tests of cast iron to be conducted at the University of Wisconsin.

The balance of income of the Research Fund on May 31, 1937, with $400 of the above grants still to be paid, was $954.27.

This year Committee E-9 will not directly address the various committees to learn if they have need of funds for their work which might be made by grants from the income of the Society's Research Fund. However, some funds are available and the committee will be pleased to consider requests for grants. These should be accompanied by a general statement of the nature of the problem under study, its need, and the reason why funds should be made available by the Research Committee.

Respectfully submitted on behalf of the committee,

P. H. BATES,
Chairman.

C. L. WARWICK,
Secretary.

June, 1937.

REPORT OF COMMITTEE E-10
ON
STANDARDS

The past year has been one of the largest in the Society's history when considered from the viewpoint of standardization activities. The appearance of the triennial Book of Standards, of course, represents a landmark in standardization activities and, looking toward its publication, the committees at the last annual meeting made a large number of recommendations with respect to the adoption of standards and the adoption of revisions of existing standards in order that the specifications and test methods as published in the Book of Standards would reflect the latest current practice. There has been continued activity during the year. The amount of attention given to the undertaking of new projects has been most impressive, indicating that there is still much to be done in the way of standardization. Probably never before in the Society's history have so many new committees been organized, for during the past year, as indicated later in the report, organization has been effected of four new committees with a fifth definitely authorized and in process of organization.

The activities of Committee E-10 on Standards during the past year are set forth under the following heads: Review of A.S.T.M. standardization activities, consideration of new and revised standards and tentative standards, promotion and expansion of standardization activities, and relations with American Standards Association.

As in the previous year, a very considerable number of new tentative standards and revisions of existing standards and tentative standards have been submitted to Committee E-10 during the year for approval. For the most part these were considered at the regular meeting of the committee held in Philadelphia, Pa., on August 26, 1936.

The Executive Committee of the Society reappointed Mr. Cloyd M. Chapman, Consulting Engineer, and Mr. H. L. Curtis of the National Bureau of Standards to serve as members of Committee E-10 for a three-year term, expiring in 1939.

REVIEW OF A.S.T.M. STANDARDIZATION ACTIVITIES

A general summary of the new tentative standards submitted at the 1936 annual meeting, of tentative standards adopted as standard and of revisions in standards was included in the July issue of the *ASTM Bulletin*, together with a review of the standardization work of the various committees with statements concerning new standards in prospect. Much of the information, particularly in respect to standardization projects under development in committees, is furnished by the standing committees subsequent to the annual meeting and takes advantage of the fact that many committees plan their next year's work at committee meetings held during the annual meeting.

It is possible to make only an approximate estimate in terms of new and revised standards of the volume of standardization work accomplished during the year and reported to the Society at this meeting. The reports of standing committees to be presented at the annual meeting include recommendations to the Society substantially as follows:

New tentative standards	51
Revisions of existing tentative standards	48
Tentative revisions of existing standards	29
Tentative standards recommended for adoption as standard	19
Revisions of existing standards recommended for adoption as standard	20
Standards and tentative standards withdrawn	34

There are at present 513 standards and 285 tentative standards of the Society, making a total of 798, and if the above recommendations are approved this total will become 815.

CONSIDERATION OF NEW AND REVISED STANDARDS

Proposed new standards approved by Committee E-10 and published as tentative during the year are as follows:

Tentative Specifications for:

Seamless Steel Boiler Tubes for High-Pressure Service (A 192 – 36 T), submitted by Committee A-1 on Steel,

Alloy-Steel Bolting Materials for High Pressure and Temperatures to 1100 F. (A 193 – 36 T), submitted by Committee A-1,

Nuts Used in Bolting for High-Pressure Service at Temperatures up to 1100 F. (A 194 – 36 T), submitted by Committee A-1,

High-Strength Structural Rivet Steel (A 195 – 36 T), submitted by Committee A-1,

Cupola Malleable Iron (A 197 – 36 T), submitted by Committee A-7 on Malleable Iron Castings,

20 per cent Chromium, 9 per cent Nickel Alloy Steel Castings (A 198 – 36 T), submitted by Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys,

Aluminum-Base Alloy Permanent Mold Castings (B 108 – 36 T), submitted by Committee B-7 on Light Metals and Alloys, Cast and Wrought,

Alluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 – 36 T), submitted by Committee B-7,

Glazed Building Units (C 126 – 36 T), submitted by Committee C-3 on Brick, and

Gasoline (D 439 – 37 T), submitted by Committee D-2 on Petroleum Products and Lubricants

Tentative Methods of:

Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 36 T), submitted by Committees C-9 on Concrete and Concrete Aggregates and D-4 on Road and Paving Materials, to replace Methods D 30 – 18 and C 86 – 31 T,

Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 36 T), submitted by Committees C-9 and D-4, to replace Methods C 68 – 30 and D 55 – 25,

Test for Softening Point by Ring-and-Ball Apparatus (E 28 – 36 T), submitted by Committee E-1 on Methods of Testing, and

Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron and Wrought Iron (E 30 – 36 T), submitted by Committee E-3 on Chemical Analysis of Metals, to replace Methods A 33 – 24, A 55 – 24, A 64 – 27 and A 130 – 30.

Tentative Definitions of:

Terms Relating to Cast Iron (A 196 – 36 T), submitted by Committee A-3 on Cast Iron.

Revisions of the following tentative standards were approved by Committee E-10:

Tentative Specifications for:

Wrought-Iron Plates (A 42 – 36 T), submitted by Committee A-2 on Wrought Iron,

Copper-Silicon Alloy Plates and Sheets (B 96 – 36 T), submitted by Committee B-5 on Copper and Copper Alloys, Cast and Wrought,

Sheet Copper-Silicon Alloy (B 97 – 36 T), submitted by Committee B-5,

Copper-Silicon Alloy Rods, Bars and Shapes (B 98 – 36 T), submitted by Committee B-5,

Copper-Silicon Alloy Wire for General Purposes (B 99 – 36 T), submitted by Committee B-5,

Aluminum Sheet and Plate (B 25 – 36 T), submitted by Committee B-7 on Light Metals and Alloys, Cast and Wrought,

Aluminum-Base Alloy Sand Castings (B 26 – 36 T), submitted by Committee B-7,

Aluminum-Alloy (Duralumin) Sheet and Plate (Aluminum-Copper-Magnesium-Manganese) (B 78 – 36 T), submitted by Committee B-7,

Aluminum-Manganese Alloy Sheet and Plate (B 79 – 36 T), submitted by Committee B-7,

Aluminum-Alloy (Duralumin) Bars, Rods and Shapes (Aluminum-Copper-Magnesium-Manganese) (B 89 – 36 T), submitted by Committee B-7,

Building Brick (Made from Clay or Shale) (C 62 – 36 T), submitted by Committee C-3 on Brick,

Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 – 36 T), submitted by Committee D-11 on Rubber Products, and

Insulated Wire and Cable: Performance Rubber Compound (D 353 – 36 T), submitted by Committee D-11.

Tentative Methods of:

Testing Brick (Modulus of Rupture, Compressive Strength, Absorption) (C 67 – 36 T), submitted by Committee C-3 on Brick,

Testing Varnishes Used for Electrical Insulation (D 115 – 36 T), submitted by Committee D-9 on Electrical Insulating Materials, and

Testing Sheet, Tape, and Molded Insulating Materials for Dielectric Strength (D 149 – 36 T), submitted by Committee D-9.

Tentative Definitions of:

Terms Relating to Petroleum (D 288 – 36 T), submitted by Committee D-2 on Petroleum Products and Lubricants.

Committee E-10 also accepted for publication as tentative a revision of the Standard Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 33), submitted by Committee D-9 on Electrical Insulating Materials.

The following standards were reverted to a tentative status on the recommendation of Committee E-3 on Chemical Analysis of Metals:

Standard Methods of:

Sampling Ferro-Alloys (A 103 – 27),
Chemical Analysis of Ferro-Alloys (A 104 – 27),
Chemical Analysis of Ferrotungsten and Ferromolybdenum (A 104 – 34),
Chemical Analysis of Alloys of Lead, Tin, Antimony and Copper (B 18 – 21),
Chemical Analysis of Manganese Bronze (B 27 – 19),
Chemical Analysis of Gun Metal (B 28 – 19),
Battery Assay of Copper (B 34 – 20),
Chemical Analysis of Pig Lead (B 35 – 24),
Chemical Analysis of Slab Zinc (Spelter) (B 38 – 21),
Chemical Analysis of Aluminum and Light Aluminum Alloys (B 40 – 35),
Chemical Analysis of Nickel (B 41 – 23),
Chemical Analysis of Brass Ingots and Sand Castings (B 45 – 27),
Chemical Analysis of Bronze Bearing Metal (B 46 – 27), and
Chemical Analysis of Silver Solders (B 81 – 33).

PROMOTION AND EXPANSION OF STANDARDIZATION ACTIVITIES

Authorization was granted by the Executive Committee for the organization of five new standing committees of the Society, as follows: Committees D-6 on Paper and Paper Products, D-18 on Soils for Engineering Purposes, C-14 on Glass and Glass Products, D-20 on Plastics, and a Committee on Thermal Insulating Materials. Committee D-18 held its organization meeting in November, 1936, and is now actively engaged in studying various methods of testing soils. The organization of the Committees on Paper, Glass, and Plastics will be effected at the present annual meeting. Progress is being made toward the formation of the Committee on Thermal Insulating Materials; formal organization may be effected during the coming year.

Considerable interest has been shown in the subject of sulfur-base jointing compounds. The possibility of having a study on these materials undertaken by one of the existing committees of the Society or whether the formation of a new standing committee would be warranted will receive the attention of Committee E-10 during the coming year.

RELATIONS WITH AMERICAN STANDARDS ASSOCIATION

Standards Submitted Under Proprietary Sponsorship Procedure.—The proprietary sponsorship procedure for submitting standards to the American Standards Association is particularly appropriate for a large number of A.S.T.M. standards. Under this procedure the following standards have been approved during the year by the A.S.A. as American standards:

Standard Specifications for:

- Carbon-Steel Castings for Valves, Flanges and Fittings for High-Temperature Service (A 95 – 36) (A.S.A. No. G17.1–1936),
- Forged or Rolled Steel Pipe Flanges for High-Temperature Service (A 105 – 36) (A.S.A. No. G17.3–1936),
- Steel for Bridges (A 7 – 36) (A.S.A. No. G18–1936),
- Steel for Buildings (A 9 – 36) (A.S.A. No. G19–1936),
- Mild Steel Plates (A 10 – 34) (A.S.A. No. G20–1936),
- Structural Rivet Steel (A 141 – 36) (A.S.A. No. G21–1936),
- Refined Iron Bars (A 41 – 36) (A.S.A. No. G12–1936)
- Uncoated Wrought-Iron Sheets (A 162 – 36) (A.S.A. No. G23–1937),
- Bronze Trolley Wire (B 9 – 36) (A.S.A. No. H22.1–1937),
- Copper Trolley Wire (B 47 – 36) (A.S.A. No. H22.2–1937),
- Zinc Oxide (D 79 – 24) (A.S.A. No. K22–1937),
- Basic Carbonate White Lead (D 81 – 34) (A.S.A. No. K23–1937),
- Red Lead (D 83 – 31) (A.S.A. No. K24–1937),
- Mineral Iron Oxide (D 84 – 27) (A.S.A. No. K25–1937),
- Lampblack (D 209 – 30) (A.S.A. No. K26–1937),
- Chrome Yellow (D 211 – 27) (A.S.A. No. K27–1937),
- Reduced Chrome Green (D 213 – 27) (A.S.A. No. K28–1937),
- Prussian Blue (D 261 – 28) (A.S.A. No. K29–1937),
- Ultramarine Blue (D 262 – 28) (A.S.A. No. K30–1937),
- Commercial Para Red (D 264 – 28) (A.S.A. No. K31–1937), and
- Standard Rules Governing the Preparation of Micrographs of Metals and Alloys, Including Recommended Practice for Photography as Applied to Metallography (E 2 – 36) (A.S.A. Z30.1–1936).

There has also been submitted to the A.S.A. for approval as American standards under the proprietary sponsorship procedure, the Standard Specifications for Spirits of Turpentine (D 13 – 34) and the Standard Methods of Sampling and Testing Turpentine (D 233 – 36). These are now receiving consideration in the A.S.A.

Standards Submitted Under Sectional Committee Procedure.—Reports have been received from several of the sectional committees for which the Society is sponsor, submitting standards to the A.S.A. for approval. These reports have been transmitted to that association and the following standards have been approved during the year:

On the Recommendation of Sectional Committee B36 on Standardization of Dimensions and Materials of Wrought-Iron and Wrought-Steel Pipe and Tubing:

Approved as American Standards:

Standard Specifications for Welded and Seamless Steel Pipe (A 53 - 36) (A.S.A. No. B36.1-1936),

Standard Specifications for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 36) (A.S.A. No. B36.3-1936),

Standard Specifications for Electric-Fusion-Welded Steel Pipe (Sizes 30 in. and over) (A 134 - 36) (A.S.A. No. B36.4-1936), and

Standard Specifications for Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not including 30 in.) (A 139 - 36) (A.S.A. No. B36.9-1936).

On the Recommendation of Sectional Committee C59 on Electrical Insulating Materials:

Approved as American Standard:

Standard Methods of Testing Electrical Insulating Oils (D 117 - 36) (A.S.A. No. C59.2-1937).

On the Recommendation of Sectional Committee G8 on Specifications for Zinc Coating of Iron and Steel:

Approved as American Standards:

Standard Specifications for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 - 36) (A.S.A. No. G8.7-1937), and

Standard Specifications for Zinc-Coated (Galvanized) Wrought-Iron Sheets (A 163 - 36) (A.S.A. No. G8.8-1937).

On the Recommendation of Sectional Committee M20 on Classification of Coals:

Approved as American Tentative Standards:

Tentative Specifications for Classification of Coals by Rank (D 388 - 36 T) (A.S.A. No. M20.1-1936), and

Tentative Specifications for Classification of Coals by Grade (D 389 - 34 T) (A.S.A. No. M20.2-1936).

On the Recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants:

Approved as American Standards:

Standard Abridged Volume Correction Table for Petroleum Oils (D 206 - 36) (A.S.A. No. Z11.1-1936),

Standard Methods of Test for Viscosity by Means of the Saybolt Viscosimeter (D 88 - 36) (A.S.A. No. Z11.2-1936),

Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester (D 93 - 36) (A.S.A. No. Z11.7-1936),

Standard Method of Test for Steam Emulsion of Lubricating Oils (D 157 - 36) (A.S.A. No. Z11.15-1936), Standard Method of Test for Burning Quality of Kerosene Oils (D 187 - 36) (A.S.A. No. Z11.17-1936),

Standard Method of Test for Burning Quality of Long-Time Burning Oil for Railway Use (D 219 - 36) (A.S.A. No. Z11.19-1936),

Standard Method of Test for Saponification Number (D 94 - 36) (A.S.A. No. Z11.20-1936),

Standard Method of Test for Flash Point by Means of the Tag Closed Tester (D 56 - 36) (A.S.A. No. Z11.24-1936),

Standard Method for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (D 189 - 36) (A.S.A. No. Z11.25-1936),

Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (D 287 - 36) (A.S.A. No. Z11.31-1936),

Standard Method of Test for Distillation of Crude Petroleum (D 285 - 36) (A.S.A. No. Z11.32-1936), and

Standard Method of Test for Gum Content of Gasoline (D 381 - 36) (A.S.A. No. Z11.36-1936).

Approved as American Tentative Standards:

Tentative Definitions of Terms Relating to Petroleum (D 288 - 36 T) (A.S.A. No. Z11.28-1936), and

Tentative Method of Test for Knock Characteristics of Motor Fuels (D 357 - 36 T) (A.S.A. No. Z11.37-1936).

Sectional Committee Organized.—A Sectional Committee on Building Code Requirements for Iron and Steel (A.S.A. Project: A 57) has been organized under the procedure of the American Standards Association. The Society is interested in this subject and has accordingly appointed as its representative on the committee, Mr. H. H. Morgan of Robert W. Hunt Co.

Respectfully submitted on behalf of the committee,

CLOYD M. CHAPMAN,
Chairman.

C. L. WARWICK,
Ex-officio Secretary.

A.S.T.M.
TENTATIVE STANDARDS

ISSUED OR REVISED, 1937

The term Tentative Standard is applied to a proposed Standard which is printed for one or more years with a view of eliciting criticism, of which the committee concerned will take due cognizance before recommending final action toward its adoption as standard.

Members of the Society and others are invited to direct written criticisms of any of these Tentative Standards to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

TENTATIVE SPECIFICATIONS

FOR

CARBON-SILICON-STEEL PLATES OF ORDINARY TENSILE RANGES FOR FUSION-WELDED BOILERS AND OTHER PRESSURE VESSELS[1]

A.S.T.M. Designation: A 201 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover carbon-silicon-steel plates, in two ordinary tensile strength ranges designated Grades A and B, intended particularly for fusion welding; for use in locomotive boiler shells, boilers for stationary service and other pressure vessels. The maximum thickness of flange quality plates to be specified under these specifications shall be 2 in.; and for ordinary firebox quality plates, 6 in. A definite silicon content is specified in order to limit the carbon to the lowest practicable amount consistent with the specified tensile strength and the thickness of the material. Welding technique is of fundamental importance and it is presupposed that welding procedure will be in accordance with approved methods.

Process

2. The steel shall be made by either or both of the following processes: open-hearth or electric-furnace.

Heat Treatment

3. (*a*) Plates over 2 in. in thickness, before being fabricated, shall be uniformly heat treated to produce grain refinement. Heat treatment involving quenching in a liquid medium is not permitted. If this treatment is not done at the rolling mill the testing shall be carried out in accordance with Paragraph (*b*).

(*b*) When a fabricator, who is equipped to properly perform the work, elects to do the required heat treatment for grain refinement he shall so indicate in his orders to the mill and he shall accept the plates on the basis of the mill tests. The plate manufacturer shall make the tests herein specified, the tension and bend test specimens to be prepared from full thickness pieces heat treated under conditions he considers appropriate for grain refinement and to give the test requirements. The manufacturer shall inform the fabricator of the procedure followed in treating the pieces at the mill for guidance in treating the plates. The required physical properties shall be determined after heat treatment of the plates on specimens prepared from pieces similarly and simultaneously treated with the plates. Pieces for two tension tests, one top and one bottom, shall be provided from each plate as rolled.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-1 on Steel.

Chemical Composition

4. The steel shall conform to the following requirements as to chemical composition:

		GRADE A	GRADE B
Carbon, max., per cent...	for plates 1 in. and under in thickness......	0.20	0.24
	for plates over 1 to 2 in., incl., in thickness.	0.24	0.27
	for plates over 2 to 4 in., incl., in thickness.	0.27	0.30
	for plates over 4 to 6 in., incl., in thickness.	0.31	0.35
Manganese, max., per cent		0.80	0.80
Phosphorus, max., per cent..	Flange	0.04	0.04
	Firebox	0.035	0.035
Sulfur, max., per cent..	Flange	0.05	0.05
	Firebox	0.04	0.04
Silicon, per cent		0.15 to 0.30	0.15 to 0.30

Ladle Analysis

5. An analysis of each melt of steel shall be made by the manufacturer to determine the percentages of the elements specified in Section 4. This analysis shall be made from a test ingot taken during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative and shall conform to the requirements specified in Section 4.

Check Analysis

6. An analysis may be made by the purchaser from a broken tension test specimen representing each plate as rolled (Note). The chemical composition thus determined shall conform to the requirements specified in Section 4.

NOTE.—The term "plate as rolled," used here and in Sections 11 (*c*) and 12 (*a*), refers to the unit plate rolled from a slab or directly from an ingot in its relation to the location and number of specimens; not to its condition.

Tension Tests

7. (*a*) The material shall conform to the following requirements as to tensile properties:

		GRADE A	GRADE B
Tensile strength, lb. per sq. in.		55 000 to 65 000	60 000 to 70 000
Yield point, min., lb. per sq. in.		0.5 tens. str.	0.5 tens. str.
Elongation in 8 in., min., per cent..	Flange....	$\frac{1\,500\,000}{\text{tens. str.}}$[a]	$\frac{1\,500\,000}{\text{tens. str.}}$[a]
	Firebox...	$\frac{1\,550\,000}{\text{tens. str.}}$[a, b]	$\frac{1\,550\,000}{\text{tens. str.}}$[a, b]
Elongation in 2 in., min., per cent[c]..	Firebox...	$\frac{1\,750\,000}{\text{tens. str.}}$[d]	$\frac{1\,750\,000}{\text{tens. str.}}$[d]

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[a] See Section 8 (*a*) and (*c*).

[b] See Paragraph (*c*).

[c] When specimen shown in Fig. 2 is used; see Section 11 (*f*).

[d] See Section 8 (*b*).

(*b*) Tensile strength only need be determined on the tension test specimen taken from the top of firebox plates, and it shall not exceed 67,000 lb. per sq. in. for Grade A, nor 72,000 lb. per sq. in. for Grade B.

(*c*) Should the rule in Paragraph (*a*) for minimum elongation in 8 in. for firebox steel give a value of less than 25 per cent for Grade A material or 23 per cent for Grade B material, the minimum allowable elongation shall be 25 and 23 per cent for Grades A and B, respectively, subject to the modifications of Section 8 (*a*).

(*d*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine.

Modifications in Elongation

8. (*a*) For material over $\frac{3}{4}$ in. in thickness, a deduction from the percentage of elongation in 8 in. specified in Section 7 (*a*) and (*c*) of 0.125 per cent shall be made for each increase of $\frac{1}{32}$ in. of the specified thickness above $\frac{3}{4}$ in., to a minimum of 21 per cent for flange steel and 22 per cent for firebox steel of Grade A; and 20 per cent for flange steel and 21 per cent for firebox steel of Grade B.

(*b*) For material over $2\frac{1}{2}$ in. in thickness, a deduction from the percentage of elongation in 2 in. specified in Section 7 (*a*) of 0.5 per cent shall be made for each increase of $\frac{1}{2}$ in. of the specified thickness above $2\frac{1}{2}$ in.

(*c*) For material $\frac{1}{4}$ in. and under in thickness, the elongation shall be measured on a gage length of 24 times the thickness of the specimen.

Bend Tests

9. The test specimen shall stand being bent cold through 180 deg. without cracking on the outside of the bent portion around a pin, the diameter of which shall have the following relation to the thickness of the specimen. When the test is made on a specimen reduced in thickness, the rolled surface shall be on the outer curve of the bend:

	DIAMETER OF PIN ABOUT WHICH SPECIMEN IS BENT (t = thickness of specimen)	
THICKNESS OF MATERIAL	GRADE A	GRADE B
Up to 1 in., incl.	$\frac{1}{2}\ t$	$1\ t$
Over 1 to $1\frac{1}{2}$ in., incl.	$1\ t$	$1\frac{1}{2}\ t$
Over $1\frac{1}{2}$ to 3 in., incl.	$1\frac{1}{2}\ t$	$2\ t$
Over 3 to 4 in., incl.	$2\ t$	$2\frac{1}{2}\ t$
Over 4 to 6 in., incl.	$2\ t$	$2\frac{1}{2}\ t$

Homogeneity Test

10. (*a*) The object of the homogeneity test, which is required for firebox steel only, is to open and render visible to the eye any seams due to failure to weld up or to interposed foreign matter, or any cavities due to gas bubbles in the ingot. The fractured surface of the test specimen shall not show any single seam or cavity more than $\frac{1}{4}$ in. in length for plates $\frac{3}{4}$ in. and under in thickness; nor more than $\frac{3}{8}$ in. in length for plates over

$\frac{3}{4}$ to 4 in., inclusive, in thickness; nor more than $\frac{1}{2}$ in. in length for plates over 4 to 6 in., inclusive, in thickness, in any of the fractures obtained in accordance with Paragraph (*b*). Each fracture shall be examined and the lengths of the seams and cavities determined, a pocket lens being used if necessary.

(*b*) The test may be made on a broken tension test specimen when an 8-in. gage length specimen is used. For plates over 2 in. in thickness, the specimen for homogeneity test shall be taken adjacent, sidewise, or lengthwise, to the top tension test specimen. For plates $\frac{3}{4}$ in. and under in thickness the specimen shall be nicked or grooved transversely, in three places, about $\frac{1}{16}$ in. deep. The first groove shall be 2 in. from the square end, and each succeeding groove on the opposite side about 2 in. from the preceding one. For plates over $\frac{3}{4}$ to $2\frac{1}{8}$ in., inclusive, in thickness the specimen may be grooved $\frac{1}{8}$ in. deep, at one place on one side. The specimens shall be broken at each groove with a hammer or press, the bending being away from the groove. For plates over $2\frac{1}{8}$ in. in thickness the specimen may be grooved at one place on both sides to leave a fracture which will include the horizontal axis of the plate and have a face at least 2 in. in depth.

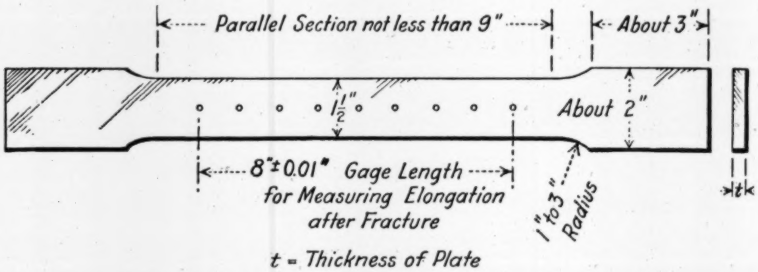
Parallel Section not less than 9"
About 3"
1½"
About 2"
8" ± 0.01" Gage Length for Measuring Elongation after Fracture
1" to 3" Radius
t
t = Thickness of Plate

FIG. 1.—Standard 8-in. Gage Length Test Specimen.

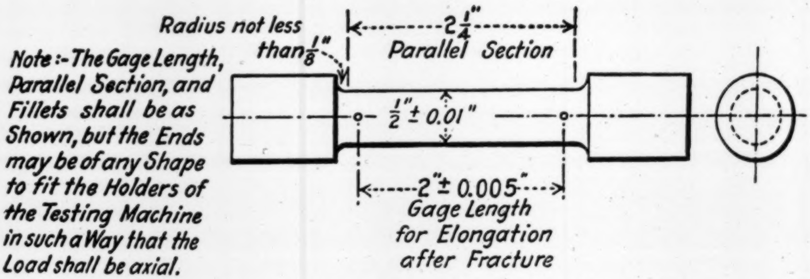
Radius not less than ⅛"
2¼" Parallel Section
Note:- The Gage Length, Parallel Section, and Fillets shall be as Shown, but the Ends may be of any Shape to fit the Holders of the Testing Machine in such a Way that the Load shall be axial.
½" ± 0.01"
2" ± 0.005" Gage Length for Elongation after Fracture

FIG. 2.—Standard 2-in. Gage Length Tension Test Specimen.

Test Specimens

11. (*a*) For plates 2 in. and under in thickness the test specimens shall be prepared for testing from the material in its rolled condition.

(*b*) For plates over 2 in. in thickness the test specimens shall be prepared from the material in its heat-treated condition, or from full thickness samples similarly and simultaneously treated. (See Section 3 (*a*) and (*b*).)

(*c*) Tension test specimens for firebox steel shall be taken from the top and bottom corners of the plate as rolled, parallel to its longitudinal axis, and for flange steel from the bottom corner of the plate only. Bend test specimens shall be taken from the middle of the top of the plate as rolled, at right angles to its longitudinal axis. (See Note under Section 6.)

(*d*) For plates 2 in. and under in thickness, tension and bend test specimens shall be the full thickness of the material and shall be machined to the form and dimensions shown in Fig. 1; or the bend test specimens may be machined with both edges parallel.

(*e*) For plates up to 4 in., inclusive, in thickness, tension test specimens may be the full thickness of the material and of the form shown in Fig. 1; when adequate testing machine capacity is available.

(*f*) For plates over 2 in. in thickness, except as permitted in Paragraph (*e*), tension test specimens shall be machined to the form and dimensions shown in Fig. 2; and the axis of each such specimen shall be located midway between the center and the top or bottom surface of the plate. The bend test specimens shall be at least $1\frac{1}{2}$ in. in width, with both edges parallel, and may be reduced to 2 in. in thickness; but shall have one surface as rolled.

(*g*) The sides of the bend test specimen may have the corners rounded to a radius of about $\frac{1}{16}$ in. for plates 2 in. and under in thickness, and to about $\frac{1}{8}$ in. for plates over 2 in. in thickness.

Number of Tests

12. (*a*) Two tension and one bend test shall be made from each firebox steel plate as rolled. One tension and one bend test shall be made from each flange steel plate as rolled. (See Note under Section 6.)

(*b*) If any test specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

(*c*) If the percentage of elongation of any tension test specimen is less than that specified in Section 7 (*a*) and any part of the fracture is more than $\frac{3}{4}$ in. from the center of the gage length of a 2-in. specimen or is outside the middle third of the gage length of an 8-in. specimen as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

Permissible Variations in Thickness and Weight

13. (*a*) The thickness of each plate shall not vary more than 0.01 in. under the thickness ordered.

(*b*) The overweight of each lot[1] of plates 2 in. and under in thickness in each shipment shall not exceed the amount given in Table I. One cubic inch of rolled steel is assumed to weigh 0.2833 lb.

TABLE I.—PERMISSIBLE OVERWEIGHTS OF PLATES ORDERED TO THICKNESS.

Ordered Thickness, in.	Permissible Excess in Average Weights per Square Foot of Plates for Widths Given, Expressed in Percentages of Nominal Weights.									Ordered Thickness, in.
	Under 48 in.	48 to 60 in., excl.	60 to 72 in., excl.	72 to 84 in., excl.	84 to 96 in., excl.	96 to 108 in., excl.	108 to 120 in., excl.	120 to 132 in., excl.	132 in. or over	
Under $\frac{1}{8}$	9	10	12	14	..	..	..	..	..	Under $\frac{1}{8}$
$\frac{1}{8}$ to $\frac{3}{16}$, excl.	8	9	10	12	..	..	..	..	..	$\frac{1}{8}$ to $\frac{3}{16}$, excl.
$\frac{3}{16}$ to $\frac{1}{4}$, excl.	7	8	9	10	12	..	..	..	..	$\frac{3}{16}$ to $\frac{1}{4}$, excl.
$\frac{1}{4}$ to $\frac{5}{16}$, excl.	6	7	8	9	10	12	14	16	19	$\frac{1}{4}$ to $\frac{5}{16}$, excl.
$\frac{5}{16}$ to $\frac{3}{8}$, excl.	5	6	7	8	9	10	12	14	17	$\frac{5}{16}$ to $\frac{3}{8}$, excl.
$\frac{3}{8}$ to $\frac{7}{16}$, excl.	4.5	5	6	7	8	9	10	12	15	$\frac{3}{8}$ to $\frac{7}{16}$, excl.
$\frac{7}{16}$ to $\frac{1}{2}$, excl.	4	4.5	5	6	7	8	9	10	13	$\frac{7}{16}$ to $\frac{1}{2}$, excl.
$\frac{1}{2}$ to $\frac{5}{8}$, excl.	3.5	4	4.5	5	6	7	8	9	11	$\frac{1}{2}$ to $\frac{5}{8}$, excl.
$\frac{5}{8}$ to $\frac{3}{4}$, excl.	3	3.5	4	4.5	5	6	7	8	9	$\frac{5}{8}$ to $\frac{3}{4}$, excl.
$\frac{3}{4}$ to 1, excl.	2.5	3	3.5	4	4.5	5	6	7	8	$\frac{3}{4}$ t 1, excl.
1 to 2, incl.	2.5	2.5	3	3.5	4	4.5	5	6	7	1 to 2, incl.

TABLE II.—PERMISSIBLE VARIATIONS OVER ORDERED THICKNESS OF PLATES OVER 2 IN. IN THICKNESS.

NOTE.—Each plate shall not vary more than 0.01 in. under the ordered thickness.

Ordered Thickness, in.	Permissible Excess in Thickness, in.					
	Width, in.					
	Up to 36, excl.	36 to 60, excl.	60 to 84, excl.	84 to 120, excl.	120 to 132, excl.	132 to 144, excl.
Over 2 to 3, excl.	0.062 ($\frac{1}{16}$)	0.094 ($\frac{3}{32}$)	0.109 ($\frac{7}{64}$)	0.125 ($\frac{1}{8}$)	0.125 ($\frac{1}{8}$)	0.141 ($\frac{9}{64}$)
3 to 4, excl.	0.078 ($\frac{5}{64}$)	0.094 ($\frac{3}{32}$)	0.109 ($\frac{7}{64}$)	0.125 ($\frac{1}{8}$)	0.125 ($\frac{1}{8}$)	0.141 ($\frac{9}{64}$)
4 to 6, excl.	0.094 ($\frac{3}{32}$)	0.125 ($\frac{1}{8}$)	0.141 ($\frac{9}{64}$)	0.141 ($\frac{9}{64}$)	0.156 ($\frac{5}{32}$)	0.172 ($\frac{11}{64}$)
6	0.109 ($\frac{7}{64}$)	0.125 ($\frac{1}{8}$)	0.156 ($\frac{5}{32}$)	0.156 ($\frac{5}{32}$)	0.172 ($\frac{11}{64}$)	....

(*c*) Each plate over 2 in. in thickness shall conform to the permissible variations over ordered thickness given in Table II.

Finish

14. The finished material shall be free from injurious defects and shall have a workmanlike finish.

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[1] The term "lot" as applied to plates 2 in. and under in thickness means all the plates of each group width and group thickness.

Marking

15. (*a*) Except as specified in Paragraph (*b*), the name or brand of the manufacturer, the manufacturer's test identification number, class, and lowest tensile strength specified in Section 7 (*a*) for the grade ordered shall be legibly stamped on each finished plate in two places not less than 12 in. from the edges and on each butt strap near the center line not less than 12 in. from each end. The manufacturer's test identification number shall be legibly stamped on each test specimen.

(*b*) For plates under $\frac{1}{4}$ in. in thickness, the marking specified in Paragraph (*a*) shall be legibly stenciled instead of stamped.

Inspection

16. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection

17. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 6, shall be reported within five working days from the receipt of samples.

(*b*) Material which shows injurious defects subsequent to its acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified.

Rehearing

18. Samples tested in accordance with Section 6, which represent rejected material, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

A.S.M.E. BOILER CONSTRUCTION CODE
SPECIFICATION No.: S-28

TENTATIVE SPECIFICATIONS
FOR
CHROME-MANGANESE-SILICON (CMS) ALLOY-STEEL PLATES FOR BOILERS AND OTHER PRESSURE VESSELS[1]

A.S.T.M. Designation: A 202 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover chrome-manganese-silicon alloy-steel plates (CMS) in two high tensile strength ranges, designated Grades A and B; up to 2 in., inclusive, in thickness; of ordinary firebox quality; for use in boilers and other pressure vessels. Grade A is a ductile material of maximum workability and Grade B a high tensile material with sufficient ductility to be workable without heating. Grade A is suitable for rivets and when so used the bars shall be subject to the requirements for rolled bars specified in the Standard Specifications for Boiler Rivet Steel and Rivets (A.S.T.M. Designation: A 31) of the American Society for Testing Materials,[2] except the chemical and physical properties.

Process

2. The steel shall be made by either or both of the following processes: open-hearth or electric-furnace.

Chemical Composition

3. The steel shall conform to the following requirements as to chemical composition:

	GRADE A	GRADE B
Carbon, max., per cent	0.17	0.25
Chromium, per cent	0.30 to 0.60	0.30 to 0.60
Manganese, per cent	1.05 to 1.40	1.05 to 1.40
Silicon, per cent	0.60 to 0.90	0.60 to 0.90
Phosphorus, max., per cent	0.035	0.035
Sulfur, max., per cent	0.04	0.04

Ladle Analysis

4. An analysis of each melt of steel shall be made by the manufacturer to determine the percentages of the elements specified in Section 3. This analysis shall be made from a test ingot taken during the pouring of the

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-1 on Steel.

[2] 1936 Book of A.S.T.M. Standards, Part I, p. 47.

melt. The chemical composition thus determined shall be reported to the purchaser or his representative, and shall conform to the requirements specified in Section 3.

Check Analysis

5. An analysis may be made by the purchaser from a broken tension test specimen representing each plate as rolled (Note). The chemical composition thus determined shall conform to the requirements specified in Section 3.

NOTE.—The term "plate as rolled," used here and in Sections 10 (*a*) and 11 (*a*), refers to the unit plate rolled from a slab or directly from an ingot in its relation to the location and number of specimens; not to its condition.

Tension Tests

6. (*a*) The material shall conform to the following requirements as to tensile properties:

	GRADE A	GRADE B
Tensile strength, lb. per sq. in.	75 000 to 90 000	85 000 to 100 000
Yield point, min., lb. per sq. in.	0.60 tens. str.	0.55 tens. str.
Elongation in 8 in., min., per cent	$\frac{1\,600\,000}{\text{tens. str.}}$[a]	$\frac{1\,600\,000}{\text{tens. str.}}$[a]

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[a] See Section 7 (*a*) and (*b*).

(*b*) Tensile strength only need be determined on the tension test specimen taken from the top of firebox plates, and it shall not exceed 92,000 lb. per sq. in. for Grade A, nor 102,000 lb. per sq. in. for Grade B.

(*c*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine.

Modifications in Elongation

7. (*a*) For plates over ¾ in. in thickness, a deduction from the percentages of elongation specified in Section 6 (*a*) of 0.125 per cent shall be made for each increase of $\frac{1}{32}$ in. of the specified thickness above ¾ in., to a minimum of 19 per cent for Grade A and 16 per cent for Grade B.

(*b*) For material ¼ in. and under in thickness, the elongation shall be measured on a gage length of 24 times the thickness of the specimen.

Bend Tests

8. The test specimen shall stand being bent cold through 180 deg. without cracking on the outside of the bent portion around a pin, the diameter of which shall have the following relation to the thickness of the specimen:

	DIAMETER OF PIN ABOUT WHICH SPECIMEN IS BENT (t = thickness of specimen)	
THICKNESS OF MATERIAL	GRADE A	GRADE B
Up to 1 in., incl.	2 t	2½ t
Over 1 to 1½ in., incl.	2½ t	3 t
Over 1½ to 2 in., incl.	3 t	3½ t

Homogeneity Test

9. (*a*) The object of the homogeneity test is to open and render visible to the eye any seams due to failure to weld up or to interposed foreign matter, or any cavities due to gas bubbles in the ingot. The fractured surface of the specimen shall not show any single seam or cavity more than $\frac{1}{4}$ in. in length for plates $\frac{3}{4}$ in. and under in thickness, nor more than $\frac{3}{8}$ in. in length for plates over $\frac{3}{4}$ to 2 in., inclusive, in thickness, in any of the fractures obtained in accordance with Paragraph (*b*). Each fracture shall be examined and the lengths of the seams and cavities determined, a pocket lens being used if necessary.

(*b*) The test may be made on a broken tension test specimen. For plates $\frac{3}{4}$ in. and under in thickness the specimen shall be nicked or grooved transversely in three places, about $\frac{1}{16}$ in. deep. The first groove shall be 2 in. from the square end, and each succeeding groove on the opposite side about 2 in. from the preceding one. The specimen shall be broken at each groove in succession with a hammer or press, the bending being away from the groove. For plates over $\frac{3}{4}$ to 2 in., inclusive, in thickness the specimen may be grooved about $\frac{1}{8}$ in. deep at one place on one side.

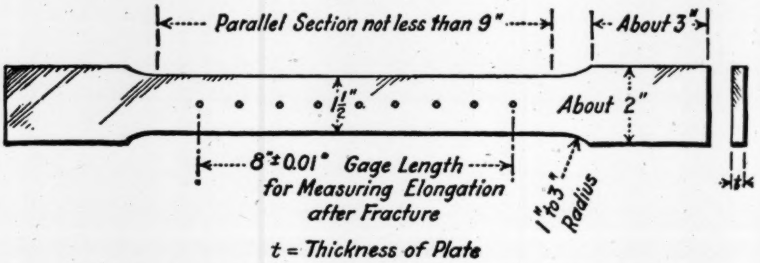
Parallel Section not less than 9"
About 3"
1½"
About 2"
8"±0.01" Gage Length for Measuring Elongation after Fracture
1" to 3" Radius
t
t = Thickness of Plate

FIG. 1.—Standard 8-in. Gage Length Test Specimen.

Test Specimens

10. (*a*) Tension test specimens shall be taken from the top and bottom corners of the plate as rolled, parallel to its longitudinal axis. Bend test specimens shall be taken from the middle of the top of the plate as rolled, at right angles to its longitudinal axis. (See Note under Section 5.)

(*b*) Tension and bend test specimens shall be of the full thickness of material as rolled, and shall be machined to the form and dimensions shown in Fig. 1.; or bend test specimens may be machined with both edges parallel. The sides of the bend test specimen may have the corners rounded to a radius of about $\frac{1}{16}$ in.

Number of Tests

11. (*a*) Two tension and one bend test shall be made from each plate as rolled. (See Note under Section 5.)

(*b*) If any test specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

(*c*) If the percentage of elongation of any tension test specimen is less than that specified in Section 6 (*a*) and any part of the fracture is outside the middle third of the gage length, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

Permissible Variations in Thickness and Weight

12. (*a*) The thickness of each plate shall not vary more than 0.01 in. under the thickness ordered.

TABLE I.—PERMISSIBLE OVERWEIGHTS OF PLATES ORDERED TO THICKNESS.

Ordered Thickness, in.	Permissible Excess in Average Weights per Square Foot of Plates for Widths Given, Expressed in Percentages of Nominal Weights.									Ordered Thickness, in.
	Under 48 in.	48 to 60 in., excl.	60 to 72 in., excl.	72 to 84 in., excl.	84 to 96 in., excl.	96 to 108 in., excl.	108 to 120 in., excl.	120 to 132 in., excl.	132 in. or over	
Under $\frac{1}{8}$	9	10	12	14	..	..	..	..	..	Under $\frac{1}{8}$
$\frac{1}{8}$ to $\frac{3}{16}$, excl.	8	9	10	12	..	..	..	..	..	$\frac{1}{8}$ to $\frac{3}{16}$, excl.
$\frac{3}{16}$ to $\frac{1}{4}$, excl.	7	8	9	10	12	..	..	..	..	$\frac{3}{16}$ to $\frac{1}{4}$, excl.
$\frac{1}{4}$ to $\frac{5}{16}$, excl.	6	7	8	9	10	12	14	16	19	$\frac{1}{4}$ to $\frac{5}{16}$, excl.
$\frac{5}{16}$ to $\frac{3}{8}$, excl.	5	6	7	8	9	10	12	14	17	$\frac{5}{16}$ to $\frac{3}{8}$, excl.
$\frac{3}{8}$ to $\frac{7}{16}$, excl.	4.5	5	6	7	8	9	10	12	15	$\frac{3}{8}$ to $\frac{7}{16}$, excl.
$\frac{7}{16}$ to $\frac{1}{2}$, excl.	4	4.5	5	6	7	8	9	10	13	$\frac{7}{16}$ to $\frac{1}{2}$, excl.
$\frac{1}{2}$ to $\frac{5}{8}$, excl.	3.5	4	4.5	5	6	7	8	9	11	$\frac{1}{2}$ to $\frac{5}{8}$, excl.
$\frac{5}{8}$ to $\frac{3}{4}$, excl.	3	3.5	4	4.5	5	6	7	8	9	$\frac{5}{8}$ to $\frac{3}{4}$, excl.
$\frac{3}{4}$ to 1, excl.	2.5	3	3.5	4	4.5	5	6	7	8	$\frac{3}{4}$ to 1, excl.
1 to 2, incl.	2.5	2.5	3	3.5	4	4.5	5	6	7	1 to 2, incl.

(*b*) The overweight of each lot[1] in each shipment shall not exceed the amount given in Table I. One cubic inch of rolled steel is assumed to weigh 0.2833 lb.

Finish

13. The finished material shall be free from injurious defects and shall have a workmanlike finish.

Marking

14. (*a*) Except as specified in Paragraph (*b*) the name or brand of the manufacturer, the manufacturer's test identification number, class, lowest tensile strength specified in Section 6 (*a*) for the grade ordered, and the letters

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[1] The term "lot" applied to Table I means all of the plates of each group width and group thickness.

"CMS" shall be legibly stamped on each finished plate in two places not less than 12 in. from the edges and on each butt strap near the center line not less than 12 in. from each end. The manufacturer's test identification number shall be legibly stamped on each test specimen.

(*b*) For plates under $\frac{1}{4}$ in. in thickness, the marking specified in Paragraph (*a*) shall be legibly stenciled instead of stamped.

Inspection

15. The inspector representing the purchaser shall have free entry at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection

16. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 5, shall be reported within five working days from the receipt of samples.

(*b*) Material which shows injurious defects subsequent to its acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified.

Rehearing

17. Samples tested in accordance with Section 5, which represent rejected material, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS

FOR

LOW-CARBON-NICKEL STEEL PLATES FOR BOILERS AND OTHER PRESSURE VESSELS[1]

A.S.T.M. Designation: A 203 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover low-carbon-nickel steel plates in three tensile strength ranges, designated Grades A, B and C; up to 2 in., inclusive, in thickness; of flange and ordinary firebox qualities; for use in locomotive boiler shells, boilers for stationary service and other pressure vessels.

Process

2. The steel shall be made by either or both of the following processes: open-hearth or electric-furnace.

Chemical Composition

3. The steel shall conform to the following requirements as to chemical composition:

		Grade A	Grade B	Grade C
Carbon, max., per cent		0.17	0.20	0.25
Manganese, max., per cent		0.80	0.80	0.80
Phosphorus, max., per cent	Flange	0.04	0.04	0.04
	Firebox	0.035	0.035	0.035
Sulfur, max., per cent	Flange	0.05	0.05	0.05
	Firebox	0.04	0.04	0.04
Silicon, per cent		0.15 to 0.30	0.15 to 0.30	0.15 to 0.30
Nickel, per cent		2.00 to 2.75	2.00 to 2.75	2.00 to 2.75

Ladle Analysis

4. An analysis of each melt of steel shall be made by the manufacturer to determine the percentages of the elements specified in Section 3. This analysis shall be made from a test ingot taken during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative, and shall conform to the requirements specified in Section 3.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-1 on Steel.

Check Analysis

5. An analysis may be made by the purchaser from a broken tension test specimen representing each plate as rolled (Note). The chemical composition thus determined shall conform to the requirements specified in Section 3.

NOTE.—The term "plate as rolled," used here and in Sections 10 (*a*) and 11 (*a*), refers to the unit plate rolled from a slab or directly from an ingot in its relation to the location and number of specimens; not to its condition.

Tension Tests

6. (*a*) The material shall conform to the following requirements as to tensile properties:

	GRADE A	GRADE B	GRADE C
Tensile strength, lb. per sq. in.	65 000 to 77 000	70 000 to 82 000	75 000 to 87 000
Yield point, lb. per sq. in., min.	0.55 tens. str.	0.55 tens. str.	0.55 tens. str.
but in no case less than	37 000	40 000	43 000
Elongation in 8 in., min., per cent: Flange	$\frac{1\ 600\ 000}{\text{tens. str.}}$[a]	$\frac{1\ 600\ 000}{\text{tens. str.}}$[a]	$\frac{1\ 600\ 000}{\text{tens. str.}}$[a]
Firebox	$\frac{1\ 650\ 000}{\text{tens. str.}}$[a]	$\frac{1\ 650\ 000}{\text{tens. str.}}$[a]	$\frac{1\ 650\ 000}{\text{tens. str.}}$[a]

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[a] See Section 7 (*a*) and (*b*).

(*b*) Tensile strength only need be determined on the tension test specimen taken from the top of firebox plates, and it shall not exceed 80,000 lb. per sq. in. for Grade A, 85,000 lb. per sq. in. for Grade B, nor 90,000 lb. per sq. in. for Grade C.

(*c*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine.

Modifications in Elongation

7. (*a*) For plates over $\frac{3}{4}$ in. in thickness, a deduction from the percentage of elongation specified in Section 6 (*a*) of 0.125 per cent shall be made for each increase of $\frac{1}{32}$ in. of the specified thickness above $\frac{3}{4}$ in. to a minimum of 21 per cent for Grade A, 20 per cent for Grade B and 19 per cent for Grade C.

(*b*) For material $\frac{1}{4}$ in. and under in thickness, the elongation shall be measured on a gage length of 24 times the thickness of the specimen.

Bend Tests

8. The test specimen shall stand being bent cold through 180 deg. without cracking on the outside of the bent portion around a pin, the diameter of which shall have the following relation to the thickness of the specimen:

THICKNESS OF MATERIAL	DIAMETER OF PIN ABOUT WHICH SPECIMEN IS BENT (t = thickness of specimen) GRADE A	GRADE B	GRADE C
Up to 1 in., incl.	$1t$	$1\frac{1}{2}t$	$2t$
Over 1 to 2 in., incl.	$1\frac{1}{2}t$	$2t$	$2\frac{1}{2}t$

Homogeneity Test

9. (*a*) The object of the homogeneity test, which is required for firebox steel only, is to open and render visible to the eye any seams due to failure to weld up or to interposed foreign matter, or any cavities due to gas bubbles in the ingot. The fractured surface of the specimen shall not show any single seam or cavity more than $\frac{1}{4}$ in. in length for plates $\frac{3}{4}$ in. and under in thickness, nor more than $\frac{3}{8}$ in. in length for plates over $\frac{3}{4}$ to 2 in., inclusive, in thickness, in any of the fractures obtained in accordance with Paragraph (*b*). Each fracture shall be examined and the lengths of the seams and cavities determined, a pocket lens being used if necessary.

(*b*) The test may be made on a broken tension test specimen. For plates $\frac{3}{4}$ in. and under in thickness the specimen shall be nicked or grooved transversely in three places, about $\frac{1}{16}$ in. deep. The first groove shall be 2 in. from the square end, and each succeeding groove on the opposite side about 2 in. from the preceding one. The specimen shall be broken at each groove in succession with a hammer or press, the bending being away from the groove. For plates over $\frac{3}{4}$ to 2 in., inclusive, in thickness the specimen may be grooved about $\frac{1}{8}$ in. deep at one place on one side.

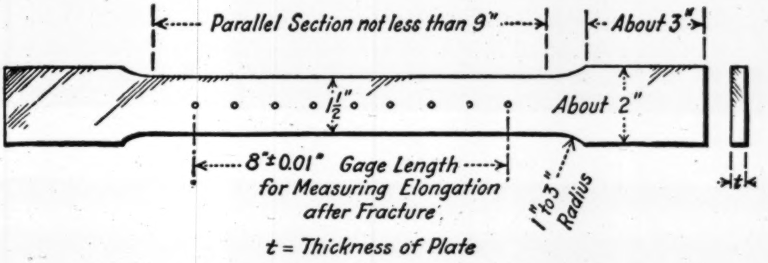
Parallel Section not less than 9"
About 3"
1½"
About 2"
8"±0.01" Gage Length for Measuring Elongation after Fracture
1" to 3" Radius
t
t = Thickness of Plate

FIG. 1.—Standard 8-in. Gage Length Test Specimen.

Test Specimens

10. (*a*) Tension test specimens for firebox material shall be taken from the top and bottom corners of the plate as rolled, parallel to its longitudinal axis, and for flange steel from the bottom corner of the plate only. Bend test specimens shall be taken from the middle of the top of the plate as rolled, at right angles to its longitudinal axis. (See Note under Section 5.)

(*b*) Tension and bend test specimens shall be of the full thickness of material as rolled, and shall be machined to the form and dimensions shown in Fig. 1; or bend test specimens may be machined with both edges parallel. The sides of the bend test specimen may have the corners rounded to a radius of about $\frac{1}{16}$ in.

Number of Tests

11. (*a*) Two tension and one bend test shall be made from each firebox steel plate as rolled. One tension and one bend test shall be made from each flange steel plate as rolled. (See Note under Section 5.)

(*b*) If any test specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

(*c*) If the percentage of elongation of any tension test specimen is less than that specified in Section 6 (*a*) and any part of the fracture is outside the middle third of the gage length, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

Permissible Variations in Thickness and Weight

12. (*a*) The thickness of each plate shall not vary more than 0.01 in. under the thickness ordered.

TABLE I.—PERMISSIBLE OVERWEIGHTS OF PLATES ORDERED TO THICKNESS.

Ordered Thickness, in.	Permissible Excess in Average Weights per Square Foot of Plates for Widths Given, Expressed in Percentages of Nominal Weights.									Ordered Thickness, in.
	Under 48 in.	48 to 60 in., excl.	60 to 72 in., excl.	72 to 84 in., excl.	84 to 96 in., excl.	96 to 108 in., excl.	108 to 120 in., excl.	120 to 132 in., excl.	132 in. or over	
Under $\frac{1}{8}$	9	10	12	14	..	..	..	..	..	Under $\frac{1}{8}$
$\frac{1}{8}$ to $\frac{3}{16}$, excl.	8	9	10	12	..	..	..	..	..	$\frac{1}{8}$ to $\frac{3}{16}$, excl.
$\frac{3}{16}$ to $\frac{1}{4}$, excl.	7	8	9	10	12	..	..	..	..	$\frac{3}{16}$ to $\frac{1}{4}$, excl.
$\frac{1}{4}$ to $\frac{5}{16}$, excl.	6	7	8	9	10	12	14	16	19	$\frac{1}{4}$ to $\frac{5}{16}$, excl.
$\frac{5}{16}$ to $\frac{3}{8}$, excl.	5	6	7	8	9	10	12	14	17	$\frac{5}{16}$ to $\frac{3}{8}$, excl.
$\frac{3}{8}$ to $\frac{7}{16}$, excl.	4.5	5	6	7	8	9	10	12	15	$\frac{3}{8}$ to $\frac{7}{16}$, excl.
$\frac{7}{16}$ to $\frac{1}{2}$, excl.	4	4.5	5	6	7	8	9	10	13	$\frac{7}{16}$ to $\frac{1}{2}$, excl.
$\frac{1}{2}$ to $\frac{5}{8}$, excl.	3.5	4	4.5	5	6	7	8	9	11	$\frac{1}{2}$ to $\frac{5}{8}$, excl.
$\frac{5}{8}$ to $\frac{3}{4}$, excl.	3	3.5	4	4.5	5	6	7	8	9	$\frac{5}{8}$ to $\frac{3}{4}$, excl.
$\frac{3}{4}$ to 1, excl.	2.5	3	3.5	4	4.5	5	6	7	8	$\frac{3}{4}$ to 1, excl.
1 to 2, incl.	2.5	2.5	3	3.5	4	4.5	5	6	7	1 to 2, incl.

(*b*) The overweight of each lot[1] in each shipment shall not exceed the amount given in Table I. One cubic inch of rolled steel is assumed to weigh 0.2833 lb.

Finish

13. The finished material shall be free from injurious defects and shall have a workmanlike finish.

Marking

14. (*a*) Except as specified in Paragraph (*b*), the name or brand of the manufacturer, the manufacturer's test identification number, class,

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[1] The term "lot" applied to Table I means all of the plates of each group width and group thickness.

lowest tensile strength specified in Section 6 (*a*) for the grade ordered, and the word "nickel" shall be legibly stamped on each finished plate in two places not less than 12 in. from the edges and on each butt strap near the center line not less than 12 in. from each end. The manufacturer's test identification number shall be legibly stamped on each test specimen.

(*b*) For plates under $\frac{1}{4}$ in. in thickness, the marking specified in Paragraph (*a*) shall be legibly stenciled instead of stamped.

Inspection

15. The inspector representing the purchaser shall have free entry at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection

16. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 5, shall be reported within five working days from the receipt of samples.

(*b*) Material which shows injurious defects subsequent to its acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified.

Rehearing

17. Samples tested in accordance with Section 5 which represent rejected material, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS

FOR

MOLYBDENUM-STEEL PLATES FOR BOILERS AND OTHER PRESSURE VESSELS[1]

A.S.T.M. Designation: A 204 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover molybdenum-steel plates, in three high tensile strength ranges, designated Grades A, B and C, intended particularly for fusion welding; for use in locomotive boiler shells, boilers for stationary service and other pressure vessels. The maximum thickness of flange quality plates to be specified under these specifications shall be 2 in.; of Grades A and B ordinary firebox quality, 6 in.; of Grade C ordinary firebox quality, 4 in. Welding technique is of fundamental importance and it is presupposed that welding procedure will be in accordance with approved methods.

Process

2. The steel shall be made by either or both of the following processes: open-hearth or electric-furnace.

Heat Treatment

3. (*a*) Plates over 2 in. in thickness, before being fabricated, shall be uniformly heat treated to produce grain refinement. Heat treatment involving quenching in a liquid medium is not permitted. If this treatment is not done at the rolling mill the testing shall be carried out in accordance with Paragraph (*b*).

(*b*) When a fabricator, who is equipped to properly perform the work, elects to do the required heat treatment for grain refinement he shall so indicate on his orders to the mill and he shall accept the plates on the basis of the mill tests. The plate manufacturer shall make the tests herein specified, the tension and bend test specimens to be prepared from full thickness pieces heat treated under conditions he considers appropriate for grain refinement and to give the test requirements. The manufacturer shall inform the fabricator of the procedure followed in treating the pieces at the mill for guidance in treating the plates. The required physical properties shall be determined after heat treatment of the plates on specimens prepared from pieces similarly and simultaneously treated with the plates. Pieces for two tension tests, one top and one bottom, shall be provided from each plate as rolled.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-1 on Steel.

Chemical Composition

4. The steel shall conform to the following requirements as to chemical composition:

		Covering Plates up to 6 in., incl., in Thickness		Covering Plates up to 4 in., incl., in Thickness
		Grade A	Grade B	Grade C
Carbon, max., per cent	for plates 1 in. and under in thickness	0.18	0.20	0.23
	for plates over 1 to 2 in., incl., in thickness	0.21	0.23	0.26
	for plates over 2 to 4 in., incl., in thickness	0.23	0.25	0.28
	for plates over 4 to 6 in., incl., in thickness	0.25	0.27	....
Manganese, per cent		0.50 to 0.90	0.50 to 0.90	0.50 to 0.90
Phosphorus, max., per cent	Flange	0.04	0.04	0.04
	Firebox	0.035	0.035	0.035
Sulfur, max., per cent	Flange	0.05	0.05	0.05
	Firebox	0.04	0.04	0.04
Silicon, per cent		0.15 to 0.30	0.15 to 0.30	0.15 to 0.30
Molybdenum, per cent		0.40 to 0.60	0.40 to 0.60	0.40 to 0.60

Ladle Analysis

5. An analysis of each melt of steel shall be made by the manufacturer to determine the percentages of the elements specified in Section 4. This analysis shall be made from a test ingot taken during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative, and shall conform to the requirements specified in Section 4.

Check Analysis

6. An analysis may be made by the purchaser from a broken tension test specimen representing each plate as rolled (Note). The chemical composition thus determined shall conform to the requirements specified in Section 4.

Note.—The term "plate as rolled," used here and in Sections 11 (*c*) and 12 (*a*), refers to the unit plate rolled from a slab or directly from an ingot in its relation to the location and number of specimens; not to its condition.

Tension Tests

7. (*a*) The material shall conform to the following requirements as to tensile properties:

		GRADE A	GRADE B	GRADE C
Tensile strength, lb. per sq. in.		65 000 to 77 000	70 000 to 82 000	75 000 to 87 000
Yield point, min., lb. per sq. in.		0.55 tens. str.	0.55 tens. str.	0.55 tens. str.
but in no case less than		37 000	40 000	43 000
Elongation in 8 in., min., per cent	Flange	$\frac{1\,600\,000}{\text{tens. str.}}$ [a]	$\frac{1\,600\,000}{\text{tens. str.}}$ [a]	$\frac{1\,600\,000}{\text{tens. str.}}$ [a]
	Firebox	$\frac{1\,650\,000}{\text{tens. str.}}$ [a]	$\frac{1\,650\,000}{\text{tens. str.}}$ [a]	$\frac{1\,650\,000}{\text{tens. str.}}$ [a]
Elongation in 2 in., min., per cent [c]	Firebox	$\frac{1\,750\,000}{\text{tens. str.}}$ [b]	$\frac{1\,750\,000}{\text{tens. str.}}$ [b]	$\frac{1\,750\,000}{\text{tens. str.}}$ [b]

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[a] See Section 8 (*a*) and (*c*).

[b] See Section 8 (*b*).

[c] When specimen shown in Fig. 2 is used; see Section 11 (*e*).

(*b*) Tensile strength only need be determined on the tension test specimen taken from the top of firebox plates, and it shall not exceed 80,000 lb. per sq. in. for Grade A, 85,000 lb. per sq. in. for Grade B, nor 90,000 lb. per sq. in. for Grade C.

(*c*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine.

Modifications in Elongation

8. (*a*) For plates over $\frac{3}{4}$ in. in thickness, a deduction from the percentage of elongation in 8 in. specified in Section 7 (*a*) of 0.125 per cent shall be made for each increase of $\frac{1}{32}$ in. of the specified thickness above $\frac{3}{4}$ in. to a minimum of 19 per cent for Grades A and B, and 18 per cent for Grade C.

(*b*) For plates over $2\frac{1}{2}$ in. in thickness, a deduction from the percentage of elongation specified in Section 7 (*a*) of 0.5 per cent shall be made for each increase of $\frac{1}{2}$ in. of the specified thickness above $2\frac{1}{2}$ in.

(*c*) For material $\frac{1}{4}$ in. and under in thickness, the elongation shall be measured on a gage length of 24 times the thickness of the specimen.

Bend Tests

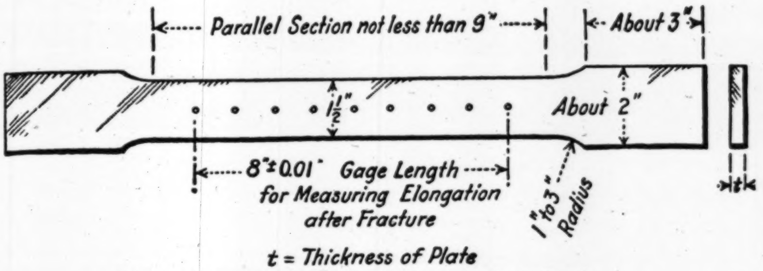
9. The test specimen shall stand being bent cold through 180 deg. without cracking on the outside of the bent portion around a pin, the diameter of which shall have the following relation to the thickness of the specimen. When the test is made on a specimen reduced in thickness, the rolled surface shall be on the outer curve of the bend:

	DIAMETER OF PIN ABOUT WHICH SPECIMEN IS BENT (t = thickness of specimen)		
THICKNESS OF MATERIAL	GRADE A	GRADE B	GRADE C
Up to 1 in., incl.	$1\,t$	$1\frac{1}{2}\,t$	$2\,t$
Over 1 to 2 in., incl.	$1\frac{1}{2}\,t$	$2\,t$	$2\frac{1}{2}\,t$
Over 2 to 4 in., incl.	$1\frac{1}{2}\,t$	$2\,t$	$3\,t$
Over 4 to 6 in., incl.	$2\,t$	$2\frac{1}{2}\,t$	....

Homogeneity Test

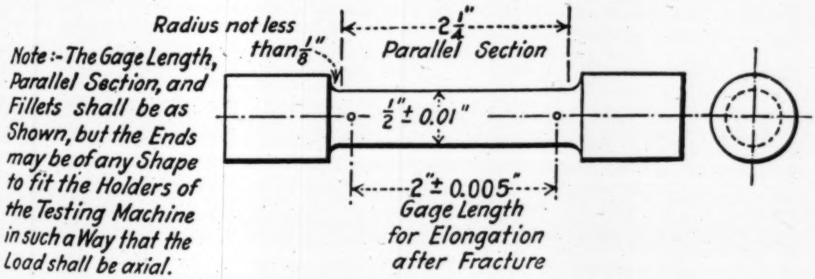
10. (*a*) The object of the homogeneity test, which is required for firebox steel only, is to open and render visible to the eye any seams due to failure to weld up or to interposed foreign matter, or any cavities due to gas bubbles in the ingot. The fractured surface of the test specimen

shall not show any single seam or cavity more than ¼ in. in length for plates ¾ in. and under in thickness; nor more than ⅜ in. in length for plates over ¾ to 4 in., inclusive, in thickness; nor more than ½ in. in length for plates over 4 to 6 in., inclusive, in thickness, in any of the fractures obtained in accordance with Paragraph (*b*). Each fracture shall be examined and the lengths of the seams and cavities determined, a pocket lens being used if necessary.


Parallel Section not less than 9"
About 3"
1½"
About 2"
8"±0.01" Gage Length for Measuring Elongation after Fracture
1" to 3" Radius
t

t = Thickness of Plate

FIG. 1.—Standard 8-in. Gage Length Test Specimen.


Radius not less than ⅛"
2¼"
Parallel Section
½" ± 0.01"
2" ± 0.005"
Gage Length for Elongation after Fracture

Note:- The Gage Length, Parallel Section, and Fillets shall be as Shown, but the Ends may be of any Shape to fit the Holders of the Testing Machine in such a Way that the Load shall be axial.

FIG. 2.—Standard 2-in. Gage Length Tension Test Specimen.

(*b*) The test may be made on a broken tension test specimen when an 8-in. gage length specimen is used. For plates over 2 in. in thickness, the specimen for homogeneity test shall be taken adjacent, sidewise, or lengthwise, to the top tension test specimen. For plates ¾ in. and under in thickness the specimen shall be nicked or grooved transversely, in three places, about $\frac{1}{16}$ in. deep. The first groove shall be 2 in. from the square end, and each succeeding groove on the opposite side about 2 in. from the preceding one. For plates over ¾ to 2⅛ in., inclusive, in thickness the specimen may be grooved ⅛ in. deep, at one place on one side. The specimens shall be broken at each groove with a hammer or press, the bending being away from the groove. For plates over 2⅛ in. in thickness the specimen may be grooved at one place on both sides to leave a fracture which will include the horizontal axis of the plate and have a face at least 2 in. in depth.

Test Specimens

11. (*a*) For plates 2 in. and under in thickness the test specimens shall be prepared for testing from the material in its rolled condition.

(*b*) For plates over 2 in. in thickness the test specimens shall be prepared from the material in its heat treated condition, or from full thickness samples similarly and simultaneously treated. (See Section 3 (*a*) and (*b*).)

(*c*) Tension test specimens for firebox steel shall be taken from the top and bottom corners of the plate as rolled, parallel to its longitudinal axis, and for flange steel from the bottom corner of the plate only. Bend test specimens shall be taken from the middle of the top of the plate as rolled, at right angles to its longitudinal axis. (See Note under Section 6.)

(*d*) For plates 2 in. and under in thickness, tension and bend test specimens shall be the full thickness of the material and shall be machined to the form and dimensions shown in Fig. 1; or the bend test specimens may be machined with both edges parallel.

(*e*) For plates over 2 in. in thickness, tension test specimens shall be machined to the form and dimensions shown in Fig. 2; and the axis of each such specimen shall be located midway between the center and the top or bottom surface of the plate. The bend test specimens shall be at least 1½ in. in width, with both edges parallel, and may be reduced to 2 in. in thickness; but shall have one surface as rolled.

(*f*) The sides of the bend test specimen may have the corners rounded to a radius of about $\frac{1}{16}$ in. for plates 2 in. and under in thickness, and to about ⅛ in. for plates over 2 in. in thickness.

Number of Tests

12. (*a*) Two tension and one bend test shall be made from each firebox steel plate as rolled. One tension and one bend test shall be made from each flange steel plate as rolled. (See Note under Section 6.)

(*b*) If any test specimen shows defective machining or develops flaws it may be discarded and another specimen substituted.

(*c*) If the percentage of elongation of any tension test specimen is less than that specified in Section 7 (*a*) and any part of the fracture is more than ¾ in. from the center of the gage length of a 2-in. specimen, or is outside the middle third of the gage length of an 8-in. specimen as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

Permissible Variations in Thickness and Weight

13. (*a*) The thickness of each plate shall not vary more than 0.01 in. under the thickness ordered.

(*b*) The overweight of each lot[1] of plates 2 in. and under in thickness in each shipment shall not exceed the amount given in Table I. One cubic inch of rolled steel is assumed to weigh 0.2833 lb.

(*c*) Each plate over 2 in. in thickness shall conform to the permissible variations over ordered thickness given in Table II.

TABLE I.—PERMISSIBLE OVERWEIGHTS OF PLATES ORDERED TO THICKNESS.

Ordered Thickness, in.	Permissible Excess in Average Weights per Square Foot of Plates for Widths Given, Expressed in Percentages of Nominal Weights.									Ordered Thickness, in.
	Under 48 in.	48 to 60 in., excl.	60 to 72 in., excl.	72 to 84 in., excl.	84 to 96 in., excl.	96 to 108 in., excl.	108 to 120 in., excl.	120 to 132 in., excl.	132 in. or over	
Under $\frac{1}{8}$	9	10	12	14	..	..	..	..	..	Under $\frac{1}{8}$
$\frac{1}{8}$ to $\frac{3}{16}$, excl.	8	9	10	12	..	..	..	..	..	$\frac{1}{8}$ to $\frac{3}{16}$, excl.
$\frac{3}{16}$ to $\frac{1}{4}$, excl.	7	8	9	10	12	..	..	..	..	$\frac{3}{16}$ to $\frac{1}{4}$, excl.
$\frac{1}{4}$ to $\frac{5}{16}$, excl.	6	7	8	9	10	12	14	16	19	$\frac{1}{4}$ to $\frac{5}{16}$, excl.
$\frac{5}{16}$ to $\frac{3}{8}$, excl.	5	6	7	8	9	10	12	14	17	$\frac{5}{16}$ to $\frac{3}{8}$, excl.
$\frac{3}{8}$ to $\frac{7}{16}$, excl.	4.5	5	6	7	8	9	10	12	15	$\frac{3}{8}$ to $\frac{7}{16}$, excl.
$\frac{7}{16}$ to $\frac{1}{2}$, excl.	4	4.5	5	6	7	8	9	10	13	$\frac{7}{16}$ to $\frac{1}{2}$, excl.
$\frac{1}{2}$ to $\frac{5}{8}$, excl.	3.5	4	4.5	5	6	7	8	9	11	$\frac{1}{2}$ to $\frac{5}{8}$, excl.
$\frac{5}{8}$ to $\frac{3}{4}$, excl.	3	3.5	4	4.5	5	6	7	8	9	$\frac{5}{8}$ to $\frac{3}{4}$, excl.
$\frac{3}{4}$ to 1, excl.	2.5	3	3.5	4	4.5	5	6	7	8	$\frac{3}{4}$ to 1, excl.
1 to 2, incl.	2.5	2.5	3	3.5	4	4.5	5	6	7	1 to 2, incl.

TABLE II.—PERMISSIBLE VARIATIONS OVER ORDERED THICKNESS OF PLATES OVER 2 IN. IN THICKNESS.

NOTE.—Each plate shall not vary more than 0.01 in. under the ordered thickness.

Ordered Thickness, in.	Permissible Excess in Thickness, in.					
	Width, in.					
	Up to 36, excl.	36 to 60, excl.	60 to 84, excl.	84 to 120, excl.	120 to 132, excl.	132 to 144, excl.
Over 2 to 3, excl.	0.062 ($\frac{1}{16}$)	0.094 ($\frac{3}{32}$)	0.109 ($\frac{7}{64}$)	0.125 ($\frac{1}{8}$)	0.125 ($\frac{1}{8}$)	0.141 ($\frac{9}{64}$)
3 to 4, excl.	0.078 ($\frac{5}{64}$)	0.094 ($\frac{3}{32}$)	0.109 ($\frac{7}{64}$)	0.125 ($\frac{1}{8}$)	0.125 ($\frac{1}{8}$)	0.141 ($\frac{9}{64}$)
4 to 6, excl.	0.094 ($\frac{3}{32}$)	0.125 ($\frac{1}{8}$)	0.141 ($\frac{9}{64}$)	0.141 ($\frac{9}{64}$)	0.156 ($\frac{5}{32}$)	0.172 ($\frac{11}{64}$)
6	0.109 ($\frac{7}{64}$)	0.125 ($\frac{1}{8}$)	0.156 ($\frac{5}{32}$)	0.156 ($\frac{5}{32}$)	0.172 ($\frac{11}{64}$)	....

Finish

14. The finished material shall be free from injurious defects and shall have a workmanlike finish.

Marking

15. (*a*) Except as specified in Paragraph (*b*), the name or brand of the manufacturer, the manufacturer's test identification number, class, lowest

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[1] The term "lot" as applied to plates 2 in. and under in thickness means all the plates of each group width and group thickness.

tensile strength specified in Section 7 (*a*) for the grade ordered, and the word "molybdenum" shall be legibly stamped on each finished plate in two places not less than 12 in. from the edges and on each butt strap near the center line not less than 12 in. from each end. The manufacturer's test identification number shall be legibly stamped on each test specimen.

(*b*) For plates under ¼ in. in thickness, the marking specified in Paragraph (*a*) shall be legibly stenciled instead of stamped.

Inspection

16. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests (except check analyses) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection

17. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 6, shall be reported within five working days from the receipt of samples.

(*b*) Material which shows injurious defects subsequent to its acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified.

Rehearing

18. Samples tested in accordance with Section 6 which represent rejected material, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS
FOR
IRON AND STEEL FILLER METAL[1]
(ARC-WELDING ELECTRODES AND GAS-WELDING RODS)

Serial Designation: A 205 – 37 T

This is a **Tentative Standard** and under the Regulations of the **Cooperating Societies** is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the **A.S.T.M.**, 260 S. Broad St., Philadelphia, **Pa.**, or of **the A.W.S., 33** W. Thirty-ninth **St., New York,** N. Y.

ISSUED, 1937.

These specifications were prepared jointly by the American Welding Society and the American Society for Testing Materials.

Scope

1. (*a*) These specifications cover arc-welding electrodes and gas-welding rods for the welding of carbon-steel shapes, plates, bars, pipe, forgings, castings and other steel products covered by the American Society for Testing Materials' specifications for material suitable for welding by the fusion process.

(*b*) The general purpose for which the filler metal is to be used shall be plainly stated in the purchase order. The statement of purpose shall include information as to the position of welding, such as flat or downhand, vertical, overhead, horizontal fillet, etc., also the welding process, such as gas, direct-current arc, alternating-current arc, or other process which the purchaser intends to use.

(*c*) Seven grades of filler metal are included, designated as Grades 2, 4, 10, 15, 20, 30, and 40, and classified in accordance with the tensile properties of the weld produced by the filler metal, as specified in Section 6 and in Table I (see Explanatory Note).

(*d*) Additional requirements (Paragraphs S1 to S8) of an optional nature are provided for use when the purchaser desires to test filler metal in the more complete manner indicated, such as when existing Code requirements must be satisfied, or the particular application justifies the expense of the additional testing. When it is desired that any of these additional requirements apply in any purchase, it shall be so stated in the order (see Explanatory Note).

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[1] Under the standardization procedure of the two Societies these specifications are under the jurisdiction of the A.S.T.M. Committee A-1 on Steel, and of the A.W.S. Filler Metal Specifications Committee.

Process

2. The filler metal may be made by any process that will fulfill the requirements of these specifications.

Standard Sizes

3. Standard sizes of filler metal are as follows: $\frac{1}{16}$, $\frac{3}{32}$, $\frac{1}{8}$, $\frac{5}{32}$, $\frac{3}{16}$, $\frac{7}{32}$, $\frac{1}{4}$, $\frac{5}{16}$, and $\frac{3}{8}$ in. in diameter. In the case of coated or covered filler metal, the standard sizes refer to the diameter of the core wire. For manual welding, standard lengths for electrodes are 14 and 18 in.; for welding rods, 36 in.

Chemical Composition

4. The filler metal shall be capable of depositing metal conforming to the following requirements as to chemical composition:

Phosphorus, max., per cent	0.04
Sulfur, max., per cent	0.04

Chemical Analysis

5. (*a*) For arc-welding electrodes, the sample for analysis shall be prepared as follows: A specimen of standard base metal (see Table I) of sufficient size, shall be prepared, and a pad of five superimposed layers of arc metal deposited thereon. The fifth or top layer shall be ground or machined away and discarded. The fourth layer shall be carefully milled off and used for the analysis specified.

(*b*) For gas-welding rods, the sample for analysis may be prepared as described in Paragraph (*a*), except that three layers shall be considered sufficient and the top layer used for the analysis, or analysis may be made of the welding rod. In the latter case, ten rods shall be selected at random from the lot, and milled in such manner as to represent the entire cross-section.

All-Weld Metal Tension Tests

6. The deposited metal shall conform to the minimum tensile properties specified in Table I.

Test Specimens

7. (*a*) From each lot of 50,000 lb., or fraction thereof, of each size of filler metal submitted for inspection, a sufficient number of electrodes or welding rods shall be selected at random from various bundles or containers, and a deposited metal specimen prepared.

(*b*) The specimen shall be prepared in the position specified in the order. If more than one position is specified, a test specimen shall be prepared in each position specified. The welding process (gas, alternating-current arc, direct-current arc, etc.) specified in the order shall be used. The welding shall be performed by a qualified welding operator,[1] following the technique recommended by the manufacturer of the filler metal.

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[1] For the requirements of a qualified welding operator, see "Tentative Rules for the Qualification of Welding Processes and Testing of Welding Operators," Am. Welding Soc., October, 1936.

(*c*) For $\frac{1}{16}$ and $\frac{3}{32}$-in. filler metal, the all-weld tension test is impracticable and the strength of these sizes shall either be judged by the strength of $\frac{1}{8}$-in. or larger filler metal or shall be determined by a joint specimen for tension test as covered in Paragraphs S3 and S4.

(*d*) For $\frac{1}{8}$-in. filler metal, a plate $\frac{3}{8}$ in. in thickness shall be prepared as illustrated in Fig. 1 and the subsize all-weld tension specimen prepared as shown.

TABLE I.—TENSILE REQUIREMENTS FOR DEPOSITED METAL.

Grade	Base Metal	Treatment of Welded Specimen[c]	All-Weld Tension Test	
			Tensile Strength, min., lb. per sq. in.	Elongation in 2 in., min., per cent
No. 2	A.S.T.M. A 70 Firebox quality steel plate,[a] tensile strength 55,000 lb. per sq. in., min.; or A.S.T.M. A 7 Steel for bridges,[b] tensile strength 60,000 lb. per sq. in., min., with carbon not over 0.25 per cent and manganese not over 1.00 per cent; or equivalent steels.	stress-relieved	80 000	20[d]
		non-stress-relieved	80 000	15[d]
No. 4	same as for Grade No. 2	stress-relieved	75 000	20[d]
		non-stress-relieved	75 000	15[d]
No. 10	same as for Grade No. 2	stress-relieved	60 000	25
		non-stress-relieved	60 000	20
No. 15	same as for Grade No. 2	stress-relieved	60 000	20
		non-stress-relieved	60 000	17
No. 20	same as for Grade No. 2	stress-relieved	52 000	10
		non-stress-relieved	55 000	7
No. 30	same as for Grade No. 2	stress-relieved	45 000	7
		non-stress-relieved	47 000	5
No. 40	same as for Grade No. 2	stress-relieved	45 000	5
		non-stress-relieved	47 000	3

[a] See Standard Specifications for Carbon-Steel Plates for Stationary Boilers and Other Pressure Vessels (A.S.T.M. Designation: A 70) of the American Society for Testing Materials, 1936 Book of A.S.T.M. Standards, Part I, p. 56.

[b] See Standard Specifications for Steel for Bridges (A.S.T.M. Designation: A 7) of the American Society for Testing Materials, 1936 Book of A.S.T.M. Standards, Part I, p. 1.

[c] Stress-relieving where called for in these specifications is for the purpose of developing the fundamental properties of the weld metal unaltered by locked-up stress. Values obtained from stress-relieved welded specimens are about 5 per cent lower in tensile strength and 10 to 20 per cent higher in ductility than those of non-stress-relieved specimens. The fact that a filler metal test requires stress-relief signifies only that it must develop the strength required regardless of stress-relief, and not that stress-relief must always be used in actual work. Stress-relieving shall be within the range of 1100 to 1200 F. for 1 hr. per 1 in. of thickness. Gas-welded specimens may be heat-treated at higher temperatures than the temperature specified above for stress-relief.

[d] It is immaterial how these high physical properties are obtained, that is, whether by carbon-steel filler metal with manganese and silicon contents or by the use of some other alloying elements, but the total amount of such other elements shall be less than 4 per cent.

(*e*) For $\frac{5}{32}$-in. and larger sizes of filler metal, a plate $\frac{3}{4}$ in. in thickness shall be prepared as illustrated in Fig. 2 and the tension specimen prepared as shown.

(*f*) Test plates shall be so supported that warping due to welding shall not throw the finished test plate out of line by an angle of over 5 deg. If plates are warped, they shall be straightened cold before being stress-relieved where required.

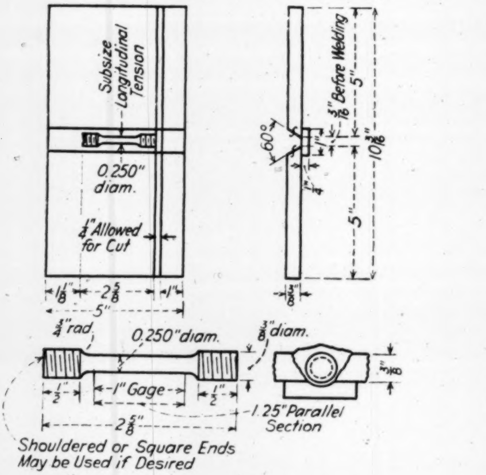
(*g*) The length of test plates may be increased as desired in order to provide material for additional tests when specified.

Retests

8. If the tension test specimen fails to conform to the requirements specified, three additional test plates shall be prepared as specified in Section 7, all of which shall conform to the requirements specified or the lot of filler metal shall be rejected.

Electrodes for Manual Welding

9. In the case of coated or covered electrodes for manual welding, about 1 in. of the electrode shall be bare or free from coating at the one end for making contact with the holder. The arc end shall be sufficiently bare to permit easy striking of the arc, but the length of the bare portion of the arc end, measured from the end to the point where the full cross-section of the coating obtains, shall not exceed one nominal diameter.


Subsize Longitudinal Tension
0.250" diam.
$\frac{1}{4}$" Allowed for Cut
$1\frac{1}{8}$"
$2\frac{5}{8}$
1"
5"
60°
$\frac{3}{16}$" Before Welding
5"
1"
$\frac{1}{4}$"
$10\frac{3}{16}$"
5"
$\frac{3}{8}$"
$\frac{3}{4}$" rad.
0.250" diam.
$\frac{3}{8}$" diam.
$\frac{1}{2}$"
1" Gage
$\frac{1}{2}$"
$\frac{3}{8}$"
$2\frac{5}{8}$"
1.25" Parallel Section
Shouldered or Square Ends May be Used if Desired

NOTE 1.—The above plate shall be used for $\frac{1}{8}$-in. filler metal.
NOTE 2.—Welds shall be deposited in either layers or beads.
NOTE 3.—Welds in vertical position shall be deposited upwards.
NOTE 4.—Electric welds shall cool to at least 100 F. between the deposition of each layer or bead.

FIG. 1.—Test Plate for $\frac{1}{8}$-in. Diameter Filler Metal and Method of Machining Specimen.

Permissible Variations in Dimensions

10. (*a*) Cross-sectional dimensions shall not vary from that specified by more than ±3 per cent.

(*b*) Cut lengths shall not vary from that specified by more than ±$\frac{1}{8}$ in.

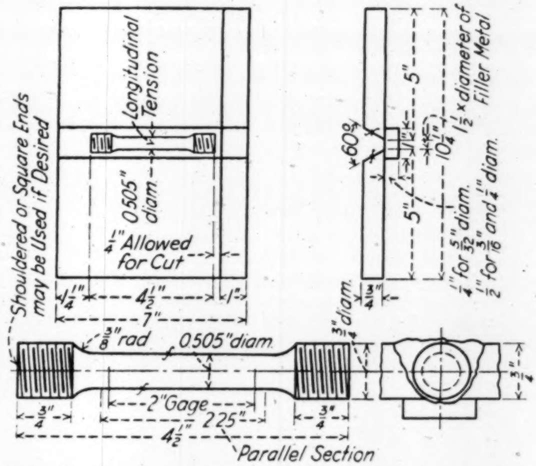
Workmanship

11. (*a*) Uncoated filler metal and the core of coated or covered filler metal shall be of uniform quality free from injurious segregation, oxides, pipe, seams or other irregularities.

(*b*) On coated or covered filler metal, the coatings and coverings shall be such that they are not readily damaged by ordinary handling and shall be of commercially uniform thickness and shall present a workmanlike appearance. Coatings and coverings shall be free from injurious scabs, blisters, abnormal pockmarks, bruises or other surface defects.

Finish

12. The surface of gas-welding rods and bare electrodes shall be smooth and free from harmful scale, oil, or grease and shall be plain or copper finished, as specified.


Shouldered or Square Ends may be Used if Desired
Longitudinal Tension
0.505" diam
$\frac{1}{4}$" Allowed for Cut
$1\frac{1}{4}$"
$4\frac{1}{2}$"
1"
7"
60°
5"
5"
$\frac{1}{2}$"
1"
$10\frac{1}{4}$"
$1\frac{1}{2}$ × diameter of Filler Metal
$\frac{1}{4}$" for $\frac{5}{32}$" diam.
$\frac{1}{2}$" for $\frac{3}{16}$" and $\frac{1}{4}$" diam.
$\frac{3}{4}$"
$\frac{3}{4}$" diam.
$\frac{3}{8}$" rad
0.505" diam.
2" Gage
2.25"
$\frac{3}{4}$"
$\frac{3}{4}$"
$4\frac{1}{2}$"
Parallel Section
$\frac{3}{4}$"

NOTE 1.—The above plate shall be used for filler metal $\frac{5}{32}$ in. in diameter and larger.
NOTE 2.—Welds shall be deposited in either layers or beads.
NOTE 3.—Welds in vertical position shall be deposited upwards.
NOTE 4.—Electric welds shall cool to at least 100 F. between the deposition of each layer or bead.

FIG. 2.—Test Plate for Filler Metal $\frac{5}{32}$ in. in Diameter and Larger and Method of Machining Specimen.

Packing

13. Filler metal shall be suitably packed, wrapped, boxed or crated to insure against injury during shipment or storage, as follows:

(*a*) Bundles of 50 or 100-lb. net weight,
(*b*) Boxes or kegs of 50, 100, 200, or 300-lb. net weight, and
(*c*) Coils or reels of approximately 50, 100, 150, 200, or 300-lb. net weight.

Marking

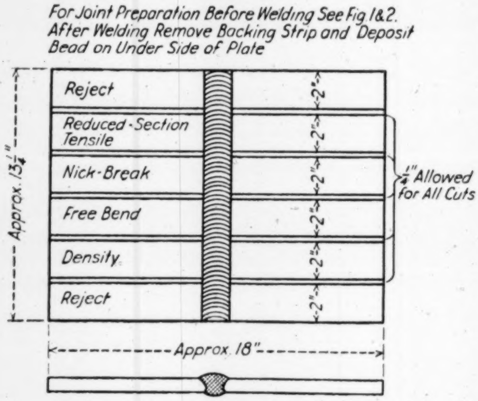
14. All bundles, boxes, kegs, coils or reels shall be legibly marked and bear the following information:

(*a*) Grade,
(*b*) Manufacturer's name and trade designation,

(*c*) Purchase order number,
(*d*) Size of filler metal, and
(*e*) Net weight.

Inspection

15. All standard tests and inspection shall be made at the place of manufacture prior to shipment, and at the manufacturer's expense, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.


For Joint Preparation Before Welding See Fig 1&2.
After Welding Remove Backing Strip and Deposit
Bead on Under Side of Plate
Reject
Reduced-Section Tensile
Nick-Break
Free Bend
Density
Reject
2"
2"
2"
2"
2"
2"
½" Allowed for All Cuts
Approx. 13¼"
Approx. 18"

NOTE

FILLER METAL, SIZE	PLATE THICKNESS
$\frac{1}{16}$ and $\frac{3}{32}$ in.	$\frac{1}{8}$ in.
$\frac{1}{8}$ in.	$\frac{3}{8}$ in.
$\frac{5}{32}$ in.	$\frac{1}{2}$ or $\frac{3}{4}$ in.

FIG. 3.—Test Plate for Additional Tests (see Paragraphs S4, S5, S6, S8).

Certification

16. When mutually agreed upon between the manufacturer and the purchaser, a certification that the material conforms to the requirements of these specifications shall be the basis of acceptance of the material.

ADDITIONAL REQUIREMENTS

S1. These additional requirements shall not be considered unless specified in the order, in which event the additional tests specified shall be made at the place of manufacture, at the purchaser's expense, and may be witnessed by his inspector before shipment of the material. The purchaser shall specify by number, the additional requirements desired.

Test Specimens for Additional Tests

S2. As specified in Section 7 (*g*), the standard test plates shown in Figs. 1 and 2 may be increased in length to furnish material for additional tests S3, S4, S5, and S8. If it is desired to prepare a separate test plate for additional tests S3, S4, S5, and S8, this shall be done using the same base-plate material as specified in Table I and following the requirements specified in Section 7. This test plate shall conform to Fig. 3, and shall be cut up for test as shown.

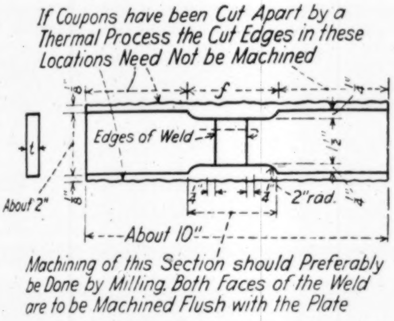

If Coupons have been Cut Apart by a Thermal Process the Cut Edges in these Locations Need Not be Machined
f
t
Edges of Weld
2
About 2"
$\frac{1}{8}$
$\frac{1}{4}$"
$\frac{1}{4}$"
2" rad.
$\frac{1}{2}$
About 10"
Machining of this Section should Preferably be Done by Milling. Both Faces of the Weld are to be Machined Flush with the Plate

FIG. 4.—Specimen for Tension Test of Welded Joint.

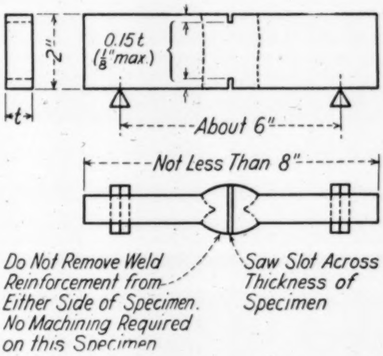

2"
0.15t
($\frac{1}{8}$" max.)
t
About 6"
Not Less Than 8"
Do Not Remove Weld Reinforcement from Either Side of Specimen. No Machining Required on this Specimen
Saw Slot Across Thickness of Specimen

FIG. 5.—Specimen for Nick-Break Test.

Tension Test of Welded Joint

S3. Test specimens of the welded joint, prepared as shown in Fig. 4 and stress-relieved unless otherwise specified, shall be tested in tension and shall conform to the following requirements:

GRADE	TENSILE STRENGTH, LB. PER SQ. IN.
No. 2	100 per cent of minimum base metal requirements
No. 4	100 per cent of minimum base metal requirements
No. 10	100 per cent of minimum base metal requirements
No. 15	100 per cent of minimum base metal requirements
No. 20	95 per cent of minimum base metal requirements
No. 30	85 per cent of minimum base metal requirements
No. 40	90 per cent of minimum base metal requirements

NOTE.—Before determining the tensile strength of the base metal, the specimen should receive the same heat treatment as is used in stress-relieving the welded test specimen.

Nick-Break Test

S4. (*a*) The nick-break test shall not be specified for Grades 30 or 40, nor for $\frac{1}{16}$ and $\frac{3}{32}$-in. filler metal of the other grades. The specimen for nick-break test of filler metal $\frac{1}{8}$ in. and larger shall be prepared as shown in Fig. 5. It shall be suitably supported as shown and broken by a sudden blow applied at the center of the weld. The blow should preferably be applied by a power hammer or falling weight, and shall be of sufficient intensity to cause a sharp, sudden fracture of the specimen through the nicked portion.

(*b*) The nick-break test for soundness of the weld shall show in the fractured surface of the specimen tested in accordance with Paragraph S4 (*a*) complete penetration through the entire thickness of the weld, absence of oxide or slag inclusions and a degree of porosity not to exceed six gas pockets per square inch of the total area of the weld surface exposed in the fracture, the maximum dimensions of any such pocket not to be in excess of $\frac{1}{16}$ in.

Free-Bend Tests

S5. (*a*) The free-bend test shall not be specified for $\frac{1}{16}$ and $\frac{3}{32}$-in. filler metal. A prepared welded-joint specimen of filler metal $\frac{1}{8}$ in. and larger shall be capable of being bent at room temperature under free-bending conditions until the least elongation measured within or across approximately the entire weld on the outside fibers of the bend test specimen conforms to the values specified in Table II. When a crack is observed in the convex surface of the specimen between the edges, the specimen shall be considered to have failed. Cracks at the corners of the specimen shall not be considered as a failure. Superficial defects which do not increase in width as the bending progresses shall not be considered as failure.

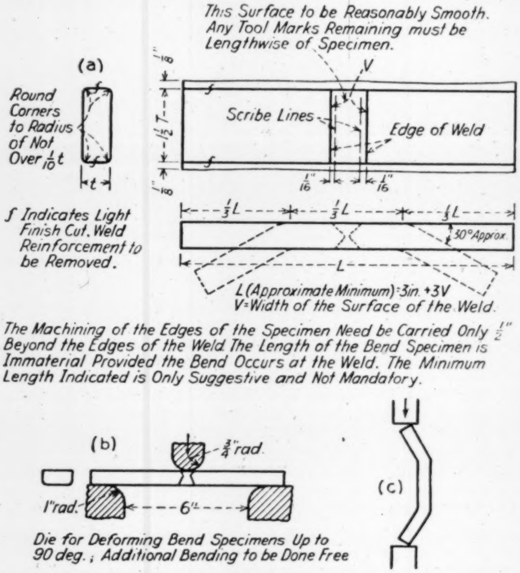

This Surface to be Reasonably Smooth. Any Tool Marks Remaining must be Lengthwise of Specimen.
(a)
Round Corners to Radius of Not Over $\frac{1}{10}$t
t
$\frac{1}{8}$"
1½ T
$\frac{1}{8}$"
V
f
f
Scribe Lines
Edge of Weld
$\frac{1}{16}$"
$\frac{1}{16}$"
f Indicates Light Finish Cut. Weld Reinforcement to be Removed.
$\frac{1}{3}$L
$\frac{1}{3}$L
$\frac{1}{3}$L
30° Approx.
L
L (Approximate Minimum) = 3in. + 3V
V = Width of the Surface of the Weld.
The Machining of the Edges of the Specimen Need be Carried Only $\frac{1}{2}$" Beyond the Edges of the Weld. The Length of the Bend Specimen is Immaterial Provided the Bend Occurs at the Weld. The Minimum Length Indicated is Only Suggestive and Not Mandatory.
(b)
$\frac{3}{4}$" rad.
1" rad.
6"
Die for Deforming Bend Specimens Up to 90 deg.; Additional Bending to be Done Free
(c)

FIG. 6.—Specimen for Free-Bend Test and Method of Testing.

(*b*) The preliminary bending of the free-bend test specimen may be performed by either of two methods, as illustrated in Fig. 6 (*a*) and (*b*). As in Fig. 6 (*a*), the bend in the specimen may be started by holding the specimen in a vise about one-third from the end, producing an initial bend at this point with hammer blows. Another bend about one-third from the other end is produced in the same manner. Or the bending of these two points may be done in a bending die with pressure rather than hammer blows. As shown in Fig. 6 (*b*), the specimen shall be placed across two supports and bent by pressure up to 90 deg. After this preliminary bending by either method, the specimen shall be placed as a strut in a vise or compression testing machine, as illustrated in Fig. 6 (*c*), and pressure applied gradually, that is, without shock, at the ends until the gage length in the weld metal on the outside of the bend has elongated the amount specified in Table II, or until failure occurs.

TABLE II.—MINIMUM JOINT REQUIREMENTS, FREE-BEND TEST.

GRADE	TREATMENT OF WELDED SPECIMEN	OUTSIDE FIBER ELONGATION, PER CENT
No. 2	Non-stress-relieved or Stress-relieved, as Specified.	..
No. 4		..
No. 10		30
No. 15		25
No. 20		20
No. 30		10
No. 40		7

Coated or Covered Electrode Requirements

S6. (*a*) The coatings or coverings on electrodes shall be capable of withstanding ordinary handling, and bending to an angle of 30 deg. about a mandrel 2 in. in diameter, without flakes falling from the coating.

(*b*) The coatings on electrodes shall not be harmfully hygroscopic. They shall not be harmed by storage under normal storage conditions for periods up to nine months.

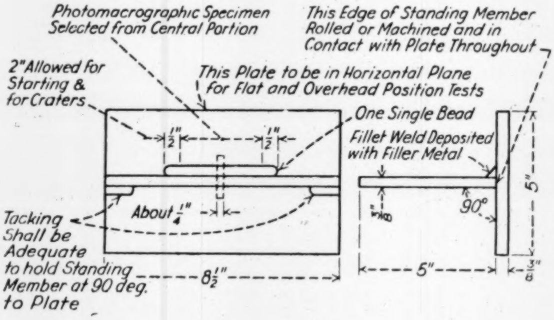
(*c*) The slag produced shall be readily removable by hand tools. Electrodes producing slag so hard as to necessitate the use of a power hammer for slag removal will not be acceptable.

(*d*) The coatings and coverings on electrodes shall be concentric to the extent that for all sizes of electrodes the maximum core-plus-one-coating dimension shall not exceed the minimum core-plus-one-coating dimension by more than 3 per cent. The concentricity shall be measured as follows:

The coating or covering shall be removed from one side of the electrode (care being taken to insure that no metal is removed from the core) at about the center of its length. The core-plus-one-coating dimension (which is the diameter of the core plus the thickness of the coating on one side) shall be measured with a micrometer. The coating shall be removed from the opposite side of the electrode at a point distant from the place where the coating was previously removed, and a similar measurement made. Two more pairs of measurements of the core-plus-one-coating dimension shall be made on planes at angles of 60 deg. and 120 deg. from the plane of the former measurements. The pair of measurements which shows the greatest percentage of variation shall determine the acceptability of the electrode.

Usability Test

S7. (*a*) Filler metal of Grades Nos. 2, 4, 10, 15, and 20 shall be capable of depositing, in all required positions, single bead fillets having a convexity ratio not greater than 30 per cent. If specified for Grades 30 and 40, the convexity ratio shall be not greater than 35 per cent. Fillet welded joints shall be made in each required position, from the same base plate material as used for all other tests, as shown by Fig. 7. Photomacrographic specimens shall then be cut, as shown in Fig. 7, from the center 6 in. of the fillet, which portion is assumed to represent the work of the filler metal; a sufficient number of specimens being taken to thoroughly represent the specimen. The region where electrodes are changed shall be excepted from examination. Photomacrographs at a magnification of four diameters shall be made of the specimens, and examined for defects.


Photomacrographic Specimen Selected from Central Portion
This Edge of Standing Member Rolled or Machined and in Contact with Plate Throughout
2" Allowed for Starting & for Craters
This Plate to be in Horizontal Plane for Flat and Overhead Position Tests
One Single Bead
1/2"
1/2"
Fillet Weld Deposited with Filler Metal
About 1/4"
Tacking Shall be Adequate to hold Standing Member at 90 deg. to Plate
90°
3/8"
5"
8 1/2"
5"
3/8"

1 Joint thus for each Required Position for $\frac{1}{8}$" and $\frac{5}{32}$" D Filler Metal
1 Joint thus in Flat Position for $\frac{3}{16}$" D and Larger Filler Metal

NOTE 1.—Fillets shall be best that welder can make after reasonable amount of practice.
NOTE 2.—Vertical welds shall be deposited upwards.
NOTE 3.—Fillets shall have the following maximum sizes:

Filler Metal Size	$\frac{1}{8}$ in.	$\frac{5}{32}$ in.	$\frac{3}{16}$ in.	$\frac{1}{4}$ in.	$\frac{5}{16}$ in.	$\frac{3}{8}$ in.
Fillet Size, max	$\frac{1}{4}$ in.	$\frac{5}{16}$ in.	$\frac{3}{8}$ in.	$\frac{1}{2}$ in.	$\frac{5}{8}$ in.	$\frac{3}{4}$ in.

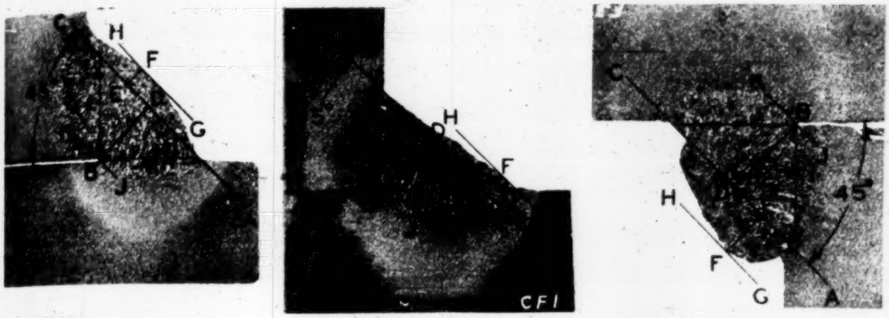
NOTE 4.—Use plates $\frac{3}{4}$ in. in thickness for tests of $\frac{5}{16}$ and $\frac{3}{8}$-in. filler metal.

FIG. 7.—Specimen for Usability Test of Filler Metal.

The convexity ratio of the fillets shall be determined as follows: On the photomacrograph a line at 45 deg. to the surface of the through plate shall be drawn internally tangent to the inmost point in the face of the fillet as shown by *A–C*, Fig. 8. A line parallel to *A–C* and tangent to the outermost point on the face of the fillet shall be drawn as shown by *G–H*; another line parallel to *A–C* and tangent to the base-and-weld-metal intersection nearest to *A–C* shall be drawn as shown by *J–K*; perpendiculars *E–F* and *D–B* shall be dropped to *A–C*, and the ratio of *E–F* to *D–B* shall be considered as the convexity ratio.

The photomacrographs shall also be examined for undercutting and overlapping.

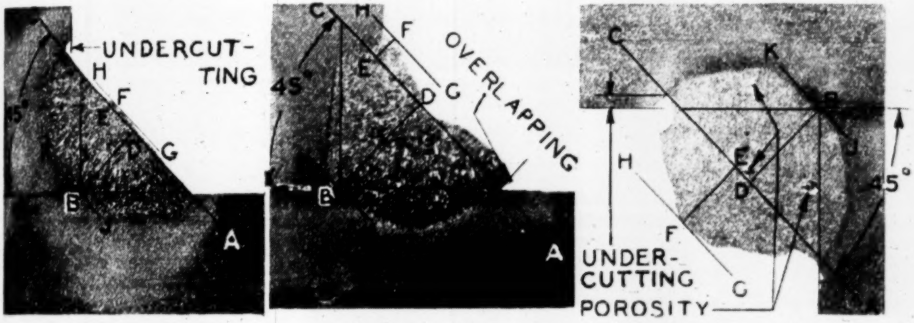
(*b*) Electrodes which undercut, that is, reduce in any degree the original thickness of the base metal, in making any required type of weld


C
H
F
G
B
J
45°
H
F
CFI
C
B
J
H
F
G
A
45°

Convexity Ratio = 0.30.........A
Undercutting..................A
Overlapping...................A
Porosity......................A
(*a*)

Convexity Ratio = 0.30.........A
Undercutting..................A
Overlapping...................A
Porosity......................A
(*b*)

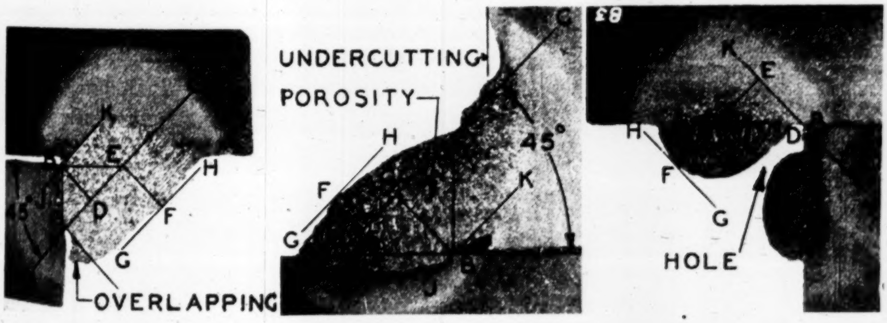
Convexity Ratio = 0.56.......NA
Undercutting..................A
Overlapping...................A
Porosity......................A
(*c*)


UNDERCUTTING
H
F
E
D
G
45°
B
A
C
H
F
45°
E
D
G
OVERLAPPING
B
A
C
K
B
J
H
E
D
45°
F
G
UNDERCUTTING
POROSITY

Convexity Ratio = 0.06.........A
Undercutting.................NA
Overlapping...................A
Porosity......................A
(*d*)

Convexity Ratio = 0.22.........A
Undercutting..................A
Overlapping..................NA
Porosity......................A
(*e*)

Convexity Ratio = 0.76.......NA
Undercutting.................NA
Overlapping...................A
Porosity.....................NA
(*f*)


K
E
H
J
45°
D
F
G
OVERLAPPING
C
UNDERCUTTING
POROSITY
H
45°
F
K
G
B
J
K
E
H
D
F
G
HOLE

Convexity Ratio = 1.15.......NA
Undercutting..................A
Overlapping..................NA
Porosity......................A
(*g*)

Convexity Ratio = 0.55.......NA
Undercutting.................NA
Overlapping...................A
Porosity.....................NA
(*h*)

Convexity Ratio = Inf........NA
Undercutting..................A
Overlapping...................A
Porosity.....................NA
(*i*)

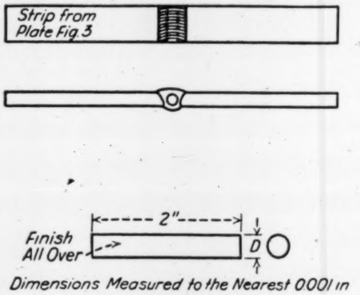
NOTE.—A. = Acceptable: NA = Unacceptable.

FIG. 8.—Acceptable and Unacceptable Fillet Welds.

Illustrations Reproduced by Courtesy of ***U. S. Navy, Bureau of Construction and Repair.***

in any required position shall not be accepted. For examples of undercutting, acceptable and non-acceptable, see Fig. 8.

(*c*) Electrodes making deposits which overlap or roll, that is, deposits which extend outside lines erected at the intersection of the base and weld metals, which lines are perpendicular to the base metal surface, in the case of bead and butt welds, and at 45 deg. to the base metal surface in the case of fillet welds, shall not be accepted. For examples of acceptable and non-acceptable overlapping, see Fig. 8.



NOTE

FILLER METAL SIZE	DIAMETER OF DENSITY SPECIMEN, D
$\frac{1}{16}$ and $\frac{1}{32}$ in.	$\frac{1}{8}$ in.
$\frac{1}{8}$ in.	$\frac{3}{8}$ in.
$\frac{5}{32}$ in. and larger	$\frac{1}{2}$ or $\frac{5}{8}$ in.

FIG. 9.—Specimen for Density Test of Filler Metal.

Density Determination

S8. The specimen for density determination shall be prepared as shown by Fig. 9. The density shall be determined by measurement and weight of the specimen, and not by the immersion method.

The results shall conform to the following minimum values:

GRADE	DENSITY (SPECIFIC GRAVITY)	GRADE	DENSITY (SPECIFIC GRAVITY)
No. 2	7.80	No. 20	7.75
No. 4	7.80	No. 30	....
No. 10	7.80	No. 40	....
No. 15	7.75		

EXPLANATORY NOTE

NOTE.—It has been most convenient to classify the several grades of filler metal in accordance with the tensile properties of the weld metal, that is, the results of so-called all-weld tension tests. Such values are probably of importance in nearly all applications of filler metal. For this reason, it has been made the basis of acceptance for general purposes. It has been clearly recognized that some fields of application, and many special cases, would require testing in a more complete manner. Also, it was appreciated that some applications would require specific requirements for coatings, and ability to deposit welds of restricted contour. To embody all of such requirements into the main body of the specifications would unduly penalize many producers and consumers alike. The most satisfactory method for handling such a situation was to include these various tests and requirements in the specifications as additional requirements of an optional nature, to be specified when and if required. There is no intent to convey the impression that they are of less importance than the all-weld tension test. The method of inclusion permits the selection of just those additional requirements that may be desired at any time.

TENTATIVE SPECIFICATIONS

FOR

SEAMLESS COLD-DRAWN INTERMEDIATE ALLOY-STEEL HEAT-EXCHANGER AND CONDENSER TUBES[1]

A.S.T.M. Designation: A 199 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. (*a*) These specifications cover several grades of chromium-molybdenum and chromium-molybdenum-silicon seamless cold-drawn intermediate alloy-steel exchanger and condenser tubes, for heat exchangers, condensers and similar heat-transfer apparatus, in sizes ½ in. to but not including 2 in. in outside diameter.

(*b*) The grade desired shall be specified on the order.

(*c*) In specifying dimensions, the purchaser shall specify either outside diameter and minimum wall thickness, or outside diameter and average wall thickness. The inside diameter shall not be specified.

Process

2. The steel shall be made by the electric-furnace process or other process approved by the purchaser.

Manufacture

3. (*a*) Tubes shall be made by the seamless process and shall be cold-drawn.

(*b*) Unless otherwise agreed upon by the manufacturer and the purchaser, tubes may be given such heat treatment as is considered desirable by the manufacturer to meet the requirements of these specifications.

Chemical Composition

4. (*a*) The steel shall conform to the following requirements as to chemical composition:

	GRADE 1	GRADE 2	GRADE 3	GRADE 4	GRADE 5
Carbon, max. per cent	0.15	0.15	0.15	0.15	0.15
Manganese, per cent	0.30 to 0.60	0.50 max.	0.40 to 0.60	0.30 to 0.60	0.30 to 0.60
Phosphorus, max. per cent	0.030	0.030	0.030	0.030	0.030
Sulfur, max. per cent	0.030	0.030	0.030	0.030	0.030
Silicon, per cent	0.50 to 1.00	0.50 to 1.00	0.45 to 0.75	0.50 max.	0.50 max.
Chromium, per cent	1.00 to 1.50	2.25 to 2.75	1.50 to 2.00	1.75 to 2.25	2.75 to 3.25
Molybdenum, per cent	0.45 to 0.65	0.45 to 0.65	0.60 to 0.80	0.45 to 0.65	0.80 to 1.00

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-1 on Steel.

(*b*) When requested by the purchaser, the manufacturer shall submit a report showing the ladle analysis of each melt of steel from which the tubes have been made.

Check Analyses

5. (*a*) When requested by the purchaser, analyses of two tubes from each melt or from each lot of 250 tubes or fraction thereof shall be made. Drillings for analysis shall be taken from several points around each tube selected for analysis. The chemical composition thus determined shall conform to the requirements specified in Section 4 (*a*).

(*b*) If the analysis of either tube does not conform to the requirements specified, analyses of two additional tubes from the same lot shall be made, each of which shall conform to the requirements specified in Section 4 (*a*).

Tension Test

6. (*a*) The material shall conform to the following minimum requirements as to tensile properties:

Tensile strength, min., lb. per sq. in.	60 000
Yield point, min., lb. per sq. in.	25 000
Elongation in 2 in., min., per cent	30

(*b*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine, or other approved method.

Flattening Test

7. (*a*) For tubes having wall thicknesses[1] not exceeding 10 per cent of their outside diameters, a section not less than 2½ in. in length shall stand being flattened between parallel plates until the distance between the plates is three times the wall thickness, without cracking or showing flaws.

(*b*) For tubes having wall thicknesses[1] exceeding 10 per cent of their outside diameters, a section not less than 2½ in. in length shall stand being flattened between parallel plates until the distance between the plates is four times the wall thickness, without cracking or showing flaws.

Flaring Test

8. (*a*) A section of tube approximately 4 in. in length shall stand being expanded at one end over a tapered mandrel having a slope of 1 in 10 (Fig. 1) until the actual average inside diameter is increased 20 per cent without cracking or showing flaws.

(*b*) *Alternative Flange Test.*—When specified in the order, a flange test may be substituted for the flaring test prescribed in Paragraph (*a*) for tubes having a wall thickness[1] less than 10 per cent of the outside diameter. In this case a section of the tube not less than 4 in. in length shall be capable of having a flange turned over at a right angle to the body of the tube without cracking or showing flaws. This flange as measured from the outside of the tube, shall not be less than $\frac{1}{8}$ in. nor more than $\frac{3}{8}$ in. Within these limits, the width of the flange shall not be less than 15 per cent of the outside diameter of the tube. It is recommended that the flaring tool and die block shown in Fig. 2 be used in making the flange test.

---

[1] The term wall thickness shall be defined as the actual mean wall of the material tested. (See Notes 1 and 2.)

(*c*) For tubes heavier than specified in Paragraph (*b*), the flange test shall not be required.

Hardness Test

9. (*a*) For all grades, a Rockwell test shall show a hardness number not to exceed 85 B Scale.

(*b*) The hardness test may be made on the tube near the end, or on a specimen cut from the tube, at the option of the manufacturer.

(*c*) In making the Rockwell hardness test, reference should be made to the Standard Methods of Rockwell Hardness Testing of Metallic Materials (A.S.T.M. Designation: E 18) of the American Society for Testing Materials.[1]

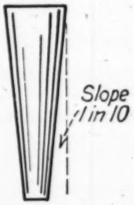

FIG. 1.—Tapered Mandrel for Flaring Test.

Hydrostatic Test

10. Each tube shall be tested at the mill to a hydrostatic pressure of 1000 lb. per sq. in.

Test Specimens

11. (*a*) Test specimens shall be taken from the ends of finished tubes before being cut to length. They shall be smooth on the ends and free from burrs and flaws.

(*b*) Tension tests shall be made in full section of the tubes.

(*c*) For tubes with upset ends, all tests specified in Sections 6, 7 and 8 shall be made on the plain end tubes, before upsetting, and the hardness test specified in Section 9 shall be made on the ends, after upsetting.

(*d*) All specimens shall be tested at room temperature.

Number of Tests

12. (*a*) From each group of 100 finished tubes or fraction thereof one tube shall be selected at random for the tension test specified in Section 6.

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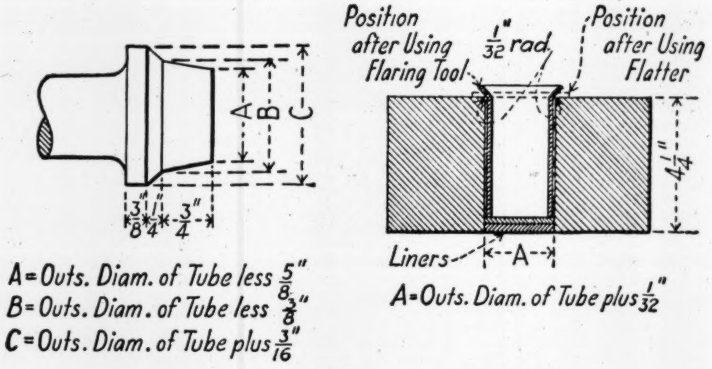
[1] 1936 Book of A.S.T.M. Standards, Part I, p. 824.

(*b*) From each heat-treated lot, one flattening and one flaring or flange test specified in Sections 7 and 8 shall be made on each of two tubes, and one hardness test specified in Section 9 shall be made on each of 5 per cent of the tubes from each heat-treated lot.

(*c*) Each tube shall be subjected to the hydrostatic test specified in Section 10.

Retests

13. (*a*) When the results of the physical tests of any group or lot do not conform to the requirements specified in Sections 6, 7, 8 and 9, retests may be made on additional tubes of double the original number from the same group or lot, each of which shall conform to the requirements specified.


Position after Using Flaring Tool
$\frac{1}{32}$" rad.
Position after Using Flatter
A
B
C
$\frac{3}{8}$"
$\frac{1}{4}$"
$\frac{3}{4}$"
$4\frac{1}{4}$"
Liners
A
A = Outs. Diam. of Tube less $\frac{5}{8}$"
B = Outs. Diam. of Tube less $\frac{3}{8}$"
C = Outs. Diam. of Tube plus $\frac{3}{16}$"
A = Outs. Diam. of Tube plus $\frac{1}{32}$"

Flaring Tool

Die Block

FIG. 2.—Flaring Tool and Die Block for Flange Test.

(*b*) If any tension test specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted. If the elongation of any tension test specimen is less than that specified, and any part of the fracture is more than $\frac{3}{4}$ in. from the center of the gage length as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

Retreatment

14. If the tubes selected to represent any group or lot fail to meet the test requirements specified in Sections 6, 7, 8 and 9, the group or lot represented may be retreated and resubmitted for test. Only two reannealing treatments shall be permitted.

Permissible Variations in Dimensions

15. Variations in specified diameter, wall thickness, and length from those specified shall not exceed the amounts shown in Table I. (See Notes 1 and 2.)

Finish

16. Finished tubes shall be reasonably straight and have smooth ends free from burrs. They shall be free from scale and injurious defects and have a workmanlike finish. Minor defects may be removed by grinding, provided the wall thickness is not decreased to less than that permitted in Section 15. A slight amount of oxidation will not be considered as scale.

Table I.—Permissible Variations in Dimensions of Tubes.

Size, Outside Diameter, in.	Outside Diameter (Including Out of Roundness), in.		Inside Diameter,[a] in.		Wall Thickness,[a] per cent		Cut Length, in.	
	Over	Under	Over	Under	Over	Under	Over	Under
Permissible Variations for Average Wall Tubes								
Under 1	0.004	0.004	0.004	0.004	10	10	$\frac{1}{8}$	0
1 to $1\frac{1}{2}$, incl	0.006	0.006	0.006	0.006	10	10	$\frac{1}{8}$	0
Over $1\frac{1}{2}$ to 2, excl	0.008	0.008	0.008	0.008	10	10	$\frac{1}{8}$	0
Permissible Variations for Minimum Wall Tubes								
Under 1	0.004	0.004	.....	.....	22	0	$\frac{1}{8}$	0
1 to $1\frac{1}{2}$, incl	0.006	0.006	.....	.....	22	0	$\frac{1}{8}$	0
Over $1\frac{1}{2}$ to 2, excl	0.008	0.008	.....	.....	22	0	$\frac{1}{8}$	0

[a] For tubes having an inside diameter less than 60 per cent of the outside diameter or having a wall $\frac{3}{4}$ in. or over in thickness, which cannot be successfully drawn over a mandrel, the inside diameter may vary over or under by an amount equal to 10 per cent of the wall thickness. The wall thickness of these tubes may vary 12.5 per cent over or under that specified.

For tubes having an inside diameter less than $\frac{1}{2}$ in., which cannot be successfully drawn over a mandrel, the wall thickness may vary 15 per cent over or under that specified; and the inside diameter will be governed by the outside diameter and wall thickness variations.

Note.—While the tubes are actually drawn to cold-drawn mechanical tubing tolerances the additional tolerances shown in Table I are required on account of warping caused by annealing and straightening.

Marking

17. The name or brand of the manufacturer, name and order number of the purchaser, grade, and hydrostatic test pressure in pounds shall be marked on a tag securely attached to the bundle or box in which the tubes are shipped.

Inspection

18. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. Unless otherwise specified, inspection and all tests shall be made at the place of manufacture.

Certification

19. When inspection at the place of manufacture has been waived by the purchaser, the manufacturer shall furnish, on request, a statement that the material has been tested and has met all the requirements of these specifications.

Rejection

20. (*a*) When inspection at the place of manufacture has been waived any rejection based on tests made in accordance with Section 5 shall be reported, by the purchaser, within five working days from receipt of samples.

(*b*) Tubes which fail in the process of installation shall be set aside and the manufacturer notified.

Rehearing

21. When inspection at place of manufacture has been waived samples, tested in accordance with Section 5, which represent rejected tubes shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests the manufacturer may make claim for a rehearing within that time.

EXPLANATORY NOTES

NOTE 1. *Average Wall.*—When an average wall tube is specified, the theoretical weight per foot of the tube is based on the exact specified dimensions. The actual wall thickness may vary over and under the specified thickness by certain definite amounts.

For the theoretical weight: $W = (D - t)t\ 10.68$
For the nominal inside diameter: $ID = D - 2t$

where W = the weight per foot of tube in pounds,
ID = the inside diameter of tube in inches,
D = the specified outside diameter of tube in inches,
t = the specified wall thickness of tube in inches, and
10.68 = the weight constant.

The actual weight of finished tubes may vary 5 per cent under or 3.5 per cent over the theoretical weight.

NOTE 2. *Minimum Wall.*—When a minimum wall tube is specified, the wall thickness shall at no point be less than that specified, but may exceed that thickness by a certain definite amount. This necessitates establishing a mean wall dimension between the maximum and minimum dimensions.

$$\text{For cold-drawn tubes the mean wall thickness} = \frac{\text{specified wall thickness}}{0.90}$$

Since the wall thickness of the tube has now been increased it follows that the weight per foot has been increased also.

The weight may be determined by the following formula:

For cold-drawn tubes: $W = (D \frac{t}{0.90}) \frac{t}{0.90} 10.68$

The actual weight of finished tubes may vary 5 per cent under or 3.5 per cent over this calculated weight.

The nominal inside diameters of minimum wall tubes may be determined by the following formula:

For cold-drawn tubes: $ID = D - 2 \frac{t}{0.90}$

where W = the weight per foot in pounds,
ID = the nominal inside diameter of tube in inches,
D = the specified outside diameter of tube in inches, and
t = the specified wall thickness of tube in inches.

TENTATIVE SPECIFICATIONS
FOR
SEAMLESS INTERMEDIATE ALLOY-STEEL STILL TUBES FOR REFINERY SERVICE[1]

A.S.T.M. Designation: A 200 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. (*a*) These specifications cover several grades of chromium-molybdenum and chromium-molybdenum-silicon intermediate alloy-steel still tubes 2 in. or over in outside diameter and heavier than No. 5 B.w.g. minimum or $\frac{1}{4}$ in. average wall thickness. These tubes are to be used for carrying oil at elevated temperatures and pressures in various types of oil stills in which the tubes may be subjected to a furnace temperature higher than that of the contained fluid.

(*b*) The grade desired shall be specified on the order.

(*c*) In specifying dimensions, the purchaser shall specify either outside diameter and minimum wall thickness, or outside diameter and average wall thickness. The inside diameter shall not be specified.

Process

2. The steel shall be made by the electric-furnace process or other process approved by the purchaser.

Manufacture

3. (*a*) Tubes shall be made by the seamless process and shall be either hot-rolled or cold-drawn, as specified.

(*b*) Unless otherwise agreed upon by the manufacturer and the purchaser, the tubes may be given such heat treatment as is considered desirable by the manufacturer to meet the requirements of these specifications.

Chemical Composition

4. (*a*) The steel shall conform to the following requirements as to chemical composition:

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-1 on Steel.

	GRADE 1	GRADE 2	GRADE 3	GRADE 4	GRADE 5
Carbon, max. per cent.	0.15	0.15	0.15	0.15	0.15
Manganese, per cent..	0.30 to 0.60	0.50 max.	0.40 to 0.60	0.30 to 0.60	0.30 to 0.60
Phosphorus, max. per cent...............	0.030	0.030	0.030	0.030	0.030
Sulfur, max. per cent..	0.030	0.030	0.030	0.030	0.030
Silicon, per cent.......	0.50 to 1.00	0.50 to 1.00	0.45 to 0.75	0.50 max.	0.50 max.
Chromium, per cent...	1.00 to 1.50	2.25 to 2.75	1.50 to 2.00	1.75 to 2.25	2.75 to 3.25
Molybdenum, per cent.	0.45 to 0.65	0.45 to 0.65	0.60 to 0.80	0.45 to 0.65	0.80 to 1.00

(*b*) The manufacturer shall submit a report showing the ladle analysis of each melt of steel from which the tubes have been made.

Check Analysis

5. (*a*) An analysis of either one billet or one tube shall be made from each melt of steel. Drillings for analysis shall be taken from several points around each tube selected for analysis. When samples for analysis are taken from the billet, they shall be taken by drilling parallel to the axis at any point midway between the outside and the center of the piece. The chemical composition thus determined shall conform to the requirements specified in Section 4 (*a*).

(*b*) If the analysis of one of these test specimens does not conform to the requirements specified in Section 4 (*a*), analysis of each billet or tube from the same melt may be made, and all billets or tubes thus complying with the requirements shall be accepted.

Tension Test

6. (*a*) The material shall conform to the following minimum requirements as to tensile properties:

Tensile strength, min., lb. per sq. in.	60 000
Yield point, min., lb. per sq. in.	25 000
Elongation in 2 in., min., per cent	30

(*b*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine, or other approved method.

Flattening Test

7. (*a*) For tubes having wall thicknesses[1] not exceeding 10 per cent of their outside diameters, a section not less than 2½ in. in length shall stand being flattened between parallel plates until the distance between the plates is three times the wall thickness, without cracking or showing flaws.

(*b*) For tubes having wall thicknesses[1] exceeding 10 per cent of their outside diameters, a section not less than 2½ in. in length shall stand being flattened between parallel plates until the distance between the plates is four times the wall thickness, without cracking or showing flaws.

Flaring Test

8. A section of tube approximately 4 in. in length shall stand being

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[1] The term wall thickness shall be defined as the actual mean wall of the material tested. (See Notes 1 and 2.)

flared with a tool having a 60-deg. included angle until the tube at the mouth of the flare has been expanded to the following percentages, without cracking or showing flaws:

Ratio of Inside Diameter to Outside Diameter[a]	Minimum Expansion of Inside Diameter, per cent
0.9	15
0.8	17
0.7	19
0.6	23
0.5	28
0.4	38
0.3	50

[a] In determining the ratio of inside diameter to outside diameter, the inside diameter shall be defined as the actual "mean inside diameter" of the material tested. (See Notes 1 and 2.)

Hardness Test

9. (*a*) For all grades, a Brinell test shall show a hardness number not to exceed 163.

(*b*) The hardness test may be made on the tube near the end or on a specimen cut from the tube, at the option of the manufacturer.

(*c*) In making the Brinell hardness test, reference should be made to the Standard Methods of Brinell Hardness Testing of Metallic Materials (A.S.T.M. Designation: E 10) of the American Society for Testing Materials.[1]

Hydrostatic Test

10. (*a*) Each tube shall be tested at the mill to a hydrostatic pressure which shall be determined by the following formula:

$$P = \frac{32{,}000\ t}{D}$$

where P = the hydrostatic test pressure in pounds per square inch,
t = the thickness of tube wall in inches,[2] and
D = the outside diameter of tube in inches.

(*b*) The maximum test pressure shall not exceed 2500 lb. per sq. in. for sizes 3 in. or under in outside diameter, nor 4500 lb. per sq. in. for sizes over 3 in. in outside diameter.

Test Specimens

11. (*a*) Test specimens shall be taken from the ends of the finished tubes before being cut to length. They shall be smooth on the ends and free from burrs and flaws.

(*b*) Tension tests, if desirable and practicable, may be made on full sections of the tubes up to the capacity of the testing machine. For larger size tubes, the tension specimen shall consist of a strip cut longitudinally from the tube and not flattened between gage marks. The sides of the specimen shall be parallel between gage marks; the width, irrespective of the thickness up to and including $\frac{3}{4}$ in., shall be 1 in.; and the gage length shall be 2 in. When the wall thickness exceeds $\frac{3}{4}$ in., the standard A.S.T.M. $\frac{1}{2}$-in. round tension test specimen may be used.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 817.

[2] The term wall thickness shall be defined as the actual mean wall of the material tested. (See Notes 1 and 2.)

(*c*) For tubes with upset ends, all tests specified in Sections 6, 7 and 8 shall be made on the plain end tubes, before upsetting, and the hardness test specified in Section 9 shall be made on the ends, after upsetting.

(*d*) All specimens shall be tested at room temperature.

Number of Tests

12. (*a*) From each group of 100 finished tubes or fraction thereof two tubes shall be selected at random for the tension test specified in Section 6.

(*b*) One flattening and one hardness test specified in Sections 7 and 9 shall be made on each end of two tubes from each heat-treated lot. A heat-treated lot consists of approximately twenty-five tubes.

(*c*) *Alternative.*—When specified in the order, the flaring test specified in Section 8 may be substituted for the flattening test and shall be made upon both ends of each of two tubes from each heat-treated lot together with an equivalent number of hardness tests specified in Section 9.

(*d*) Each tube shall be subjected to the hydrostatic test specified in Section 10.

Retests

13. (*a*) When the results of the physical tests of any group or lot do not conform to the requirements specified in Sections 6, 7, 8 and 9 (see Section 12 (*c*)), retests may be made on additional tubes of double the original number from the same group or lot, each of which shall conform to the requirements specified.

(*b*) If any tension test specimen shows defective machining or develops flaws it may be discarded and another specimen substituted.

(*c*) If the elongation of any tension test specimen is less than that specified, and any part of the fracture is more than $\frac{3}{4}$ in. from the center of the gage length as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

Retreatment

14. If individual tubes or the tubes selected to represent any group or lot fail to meet the test requirements specified in Sections 6, 7, 8 and 9, the individual tubes or the group or lot represented may be retreated and resubmitted for test. Only two reannealing treatments will be permitted.

Permissible Variations in Dimensions and Weights

15. (*a*) Variations in specified diameter, wall thickness, weight, and length shall not exceed the amounts shown in Tables I to III. (See Notes 1 and 2.)

(*b*) When requested by the purchaser, 10 per cent of the finished tubes may be weighed individually or in convenient lots.

(*c*) Tubes shall be of such diameter that a ring gage approximately 1 in. in length with a bore 0.025 in. larger than the specified outside diameter will pass over each end for a distance of 8 in. This permissible variation applies also to the ends of upset tubes.

(*d*) Permissible variations for hot-rolled tubes shall apply to the upset ends of both hot-rolled and cold-drawn tubes.

TABLE I.—PERMISSIBLE VARIATIONS IN DIMENSIONS AND WEIGHTS OF STILL TUBES.

Size	Permissible Variations											
					When Wall Thickness is Specified as "Minimum"				When Wall Thickness is Specified as "Average"			
Outside Diameter, in.	Outside Diameter, in.		Cut Length, in.		Specified Wall Thickness, per cent		Calculated Weight per Foot, per cent		Specified Wall Thickness, per cent		Theoretical Weight per Foot, per cent	
	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under
HOT-ROLLED STILL TUBES												
4 or under............	$\frac{1}{64}$	$\frac{1}{32}$	$\frac{3}{16}$	0	28	0	3.5	5	12.5	12.5	3.5	5
Over 4 to $7\frac{1}{2}$, inclusive	$\frac{1}{64}$	$\frac{3}{64}$	$\frac{3}{16}$	0	28	0	3.5	5	12.5	12.5	3.5	5
COLD-DRAWN STILL TUBES[a]												
4 or under............	0.015	0.015	$\frac{3}{16}$	0	22	0	3.5	5	10	10	3.5	5
Over 4 to $7\frac{1}{2}$, inclusive	0.015	0.025	$\frac{3}{16}$	0	22	0	3.5	5	10	10	3.5	5

[a] When cold-drawn tubes as ordered require wall thicknesses $\frac{3}{4}$ in. or over, or an inside diameter 60 per cent or less of the outside diameter, the permissible variations in wall thickness for hot-rolled tubes shown in the upper table shall apply.

Finish

16. (*a*) Finished tubes shall be reasonably straight and have smooth ends, free from burrs. They shall be free from injurious defects and have a workmanlike finish. Minor defects may be removed by grinding, provided the wall thickness is not decreased to less than that permitted in Section 15.

(*b*) The normal rotary straightening operation shall be deemed sufficient to remove scale for inspection.

Marking

17. (*a*) Tubes shall be marked, by paint stenciling, with the name or brand of the manufacturer, name and order number of the purchaser, grade, size, and hydrostatic test pressure in pounds, and whether hot-rolled or cold-drawn.

(*b*) When required by the purchaser, and if practicable, the melt number and grade of steel shall be stamped on each tube with steel stamps not less than 18 in. from one end.

Table II.—Plug Gage Dimensions for Tubes With Plain Ends.

A plug gage 4 in. in length, with a diameter determined by the proper formula given in this table shall enter each end of each tube for a distance of 4 in.

	Outside Diameter, in.	Hot-Rolled Still Tubes	Cold-Drawn Still Tubes
		Formulas[a]	Formulas[a]
When wall thickness is specified as "Minimum"........................	4 or under...............	$PG=D-\frac{2t}{0.875}-0.077$	$PG=D-\frac{2t}{0.90}-0.0625$
	Over 4 to $7\frac{1}{2}$, inclusive...	$PG=D-\frac{2t}{0.875}-0.102$	$PG=D-\frac{2t}{0.90}-0.080$
When wall thickness is specified as "Average"........................	4 or under...............	$PG=D-2t-0.077$	$PG=D-2t-0.0625$
	Over 4 to $7\frac{1}{2}$, inclusive...	$PG=D-2t-0.102$	$PG=D-2t-0.080$

[a] Expressions used in formulas are as follows:
PG = the diameter of plug gage in inches,
D = the specified outside diameter of tube in inches, and
t = the specified thickness of tube wall in inches.

Table III.—Plug Gage Dimensions for Tubes with Internal Upset Ends.

For tubes which are upset at the ends, a plug gage with a diameter determined by the proper formula given in this table shall enter the upset ends of each tube for a distance equal to the length of the upset.

Length of Upset, in.	Hot-Rolled Still Tubes		Cold-Drawn Still Tubes	
	Formulas[a]		Formulas[a]	
	Minimum Wall	Average Wall	Minimum Wall	Average Wall
3 or under..	$PG=D-2\left(\frac{t}{0.875}+U\right)-0.093$	$PG=D-2(t+U)-0.093$	$PG=D-2\left(\frac{t}{0.90}+U\right)-0.093$	$PG=D-2(t+U)-0.093$
Over 3 to 4, inclusive..	$PG=D-2\left(\frac{t}{0.875}+U\right)-0.113$	$PG=D-2(t+U)-0.113$	$PG=D-2\left(\frac{t}{0.90}+U\right)-0.113$	$PG=D-2(t+U)-0.113$
Over 4 to 5, inclusive..	$PG=D-2\left(\frac{t}{0.875}+U\right)-0.133$	$PG=D-2(t+U)-0.133$	$PG=D-2\left(\frac{t}{0.90}+U\right)-0.133$	$PG=D-2(t+U)-0.133$

[a] Expressions used in formulas are as follows:
PG = the diameter of plug gage in inches,
D = the specified outside diameter of tube in inches,
t = the specified thickness of tube wall in inches, and
U = the increase in tube wall thickness at upset end in inches.

Inspection

18. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. Unless otherwise specified, inspection and all tests shall be made at the place of manufacture.

Certification

19. When inspection at the place of manufacture has been waived by the purchaser, the manufacturer shall furnish, on request, a statement that the material has been tested and has met all the requirements of these specifications.

Rejection

20. (*a*) When inspection at place of manufacture has been waived, any rejection based on tests made in accordance with Section 5 shall be reported, by the purchaser, within five working days from receipt of samples.

(*b*) Tubes which fail in the process of installation shall be set aside and the manufacturer notified.

Rehearing

21. When inspection at place of manufacture has been waived, samples tested in accordance with Section 5 which represent rejected tubes, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

EXPLANATORY NOTES

NOTE 1: *Average Wall.*—When an average wall tube is specified, the theoretical weight per foot of the tube is based on the exact specified dimensions. The actual wall thickness may vary over and under the specified thickness by certain definite amounts.

For the theoretical weight: $W = (D - t)t\ 10.68$
For the nominal inside diameter: $ID = D - 2t$

where W = the weight per foot of tube in pounds,
ID = the inside diameter of tube in inches,
D = the specified outside diameter of tube in inches,
t = the specified wall thickness of tube in inches, and
10.68 = the weight constant.

The actual weight of finished tubes may vary 5 per cent under or 3.5 per cent over the theoretical weight.

NOTE 2: *Minimum Wall.*—When a minimum wall tube is specified, the wall thickness shall at no point be less than that specified, but may exceed that thickness by a certain definite amount. This necessitates establishing a mean wall dimension between the maximum and minimum dimensions.

$$\text{For hot-rolled tubes the mean wall thickness} = \frac{\text{specified wall thickness}}{0.875}$$

$$\text{For cold-drawn tubes the mean wall thickness} = \frac{\text{specified wall thickness}}{0.90}$$

Since the wall thickness of the tube has now been increased it follows that the weight per foot has been increased also.

The weight may be determined by the following formulas:

$$\text{For hot-rolled tubes: } W = \left(D - \frac{t}{0.875}\right) \frac{t}{0.875}\ 10.68$$

$$\text{For cold-drawn tubes: } W = \left(D - \frac{t}{0.90}\right) \frac{t}{0.90}\ 10.68$$

The actual weight of finished tubes may vary 5 per cent under or 3.5 per cent over this calculated weight.

The nominal inside diameters of minimum wall tubes may be determined by the following formulas:

$$\text{For hot-rolled tubes: } ID = D - 2\,\frac{t}{0.875}$$

$$\text{For cold-drawn tubes: } ID = D - 2\,\frac{t}{0.90}$$

where W = the weight per foot in pounds,

ID = the nominal inside diameter of tube in inches,

D = the specified outside diameter of tube in inches, and

t = the specified wall thickness of tube in inches.

TENTATIVE SPECIFICATIONS
FOR
ALLOY-STEEL BOLTING MATERIALS FOR HIGH-TEMPERATURE SERVICE FROM 750 TO 1100 F., METAL TEMPERATURES[1]

A.S.T.M. Designation: A 193 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1936; REVISED, 1937.[2]

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

Scope

1. (*a*) These specifications cover alloy-steel bolting material for pressure vessels, valves, flanges and fittings for high-temperature service. The term "bolting material" as used in these specifications covers rolled, forged or cold-drawn bars, and bolts, screws, and studs.

(*b*) Several grades of ferritic and austenitic materials are covered which are classified in accordance with their chemical and physical properties as specified in Sections 6 and 9 (*a*) (see Explanatory Note 1). Selection from the several steels should be made on the basis of the requirements of design, service conditions, physical properties, and high-temperature characteristics.

(*c*) As these steels are not necessarily all suitable for the full range of temperature up to 1100 F., care should be exercised in making the selection (see Explanatory Note 2).

Certification of Test for Bolts, Screws and Studs

2. When mutually agreed upon in writing between the manufacturer and the purchaser, a certification that the material conforms to the requirements of these specifications shall be the basis of acceptance of the material. Otherwise, the manufacturer shall report to the purchaser or his representative the results of the chemical analyses and physical tests made in accordance with these specifications.

Process

3. The steel shall be made by one or more of the following processes: open-hearth, electric-furnace or crucible.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-1 on Steel.

[2] Revision accepted by Committee E-10 on Standards, August 26, 1937.

Discard

4. A sufficient discard shall be made from each ingot to secure freedom from injurious piping and undue segregation.

Heat Treatment

5. (*a*) Ferritic steels shall be properly heat treated. The temperature for tempering or drawing back shall be at least 100 deg. Fahr. higher than the proposed operating temperature to which the material will be subjected in service.

(*b*) For austenitic steels, heat treatment shall consist of allowing the material immediately after rolling or forging to cool at a rate sufficiently rapid to prevent injury to the material; reheating to the proper temperature and holding at that temperature for a sufficient time, followed by rapid air cooling or by quenching in a liquid medium.

Chemical Composition

6. Each alloy[a] shall conform to the following requirements as to chemical composition, or to other chemical requirements as specified in the purchase order:

Class	Ferritic Steels					
Identification Symbol	B4	B5	B6	B7	B7a	B11
Grade	Nickel-Chromium-Molybdenum	4 to 6 per cent Chromium	13 per cent Chromium	Chromium-Molybdenum	Chromium-High-Molybdenum	Tungsten-Chromium-Vanadium
Carbon, per cent	0.35 to 0.45	0.35 max.	0.12 max.	0.35 to 0.45	0.35 to 0.45	0.40 to 0.50
Manganese, per cent	0.50 to 0.80	0.30 to 0.50	0.60 max.	0.60 to 0.90	0.60 to 0.90	0.20 to 0.40
Phosphorus, max., per cent	0.04	0.03	0.140^b	0.04	0.04	0.04
Sulfur, max., per cent	0.05	0.03	0.500^b	0.05	0.05	0.04
Silicon, per cent	......	0.50 max.	0.50 max.	0.15 to 0.25	0.15 to 0.25	0.15 to 0.30
Nickel, per cent	1.50 to 2.00	......	......	......	......	......
Chromium, per cent	0.50 to 0.80	4.00 to 6.00	11.5 to 13.0	0.80 to 1.10	0.80 to 1.10	1.00 to 1.50
Molybdenum, per cent	0.30 to 0.40	0.45 to 0.65^c	......	0.15 to 0.25	0.45 to 0.65	......
Tungsten, per cent	......	0.75 to 1.25^c	......	......	......	1.70 to 2.30
Vanadium, per cent	......	......	......	......	......	0.20 to 0.30

Class	Ferritic Steels				Austenitic Steel
Identification Symbol	B12	B13	B14	B15	B8
Grade	Nickel-Chromium	Tungsten-Molybdenum-Chromium	Chromium-Molybdenum-Vanadium	Silicon-Chromium-Molybdenum	18 Chromium 8 Nickel
Carbon, per cent	0.35 to 0.45	0.30 to 0.40	0.35 to 0.50	0.40 to 0.50	0.07 max.
Manganese, per cent	0.60 to 0.90	0.60 to 0.90	0.40 to 0.70	0.40 to 0.70	0.20 to 0.70
Phosphorus, max., per cent	0.04	0.04	0.04	0.03	0.140^b
Sulfur, max., per cent	0.05	0.04	0.05	0.03	0.500^b
Silicon, per cent	......	0.15 to 0.30	0.15 to 0.30	0.50 to 0.80	0.75 max.
Nickel, per cent	1.00 to 1.50	......	......	......	7.0 to 10.0
Chromium, per cent	0.45 to 0.75	0.45 to 0.75	0.80 to 1.10	1.00 to 1.50	17.0 to 20.0
Molybdenum, per cent	......	0.40 to 0.65	0.30 to 0.40	0.40 to 0.60	......
Tungsten, per cent	......	0.85 to 1.35	......	......	......
Vanadium, per cent	......	......	0.20 to 0.30	......	......

[a] As these steels are not necessarily all suitable for the full range of temperature, up to 1100 F., care should be exercised in making the selection.

[b] Where phosphorus exceeds 0.045 per cent, sulfur shall not exceed 0.050 per cent.
Where sulfur exceeds 0.050 per cent, phosphorus shall not exceed 0.045 per cent.
These limits of phosphorus and sulfur shall apply unless otherwise specified.

[c] Either molybdenum or tungsten shall be used as specified.

Ladle Analyses

7. An analysis of each melt of steel shall be made by the steel manufacturer to determine the percentages of the specified elements present. This analysis shall be made from a test ingot taken during the pouring of the melt. The chemical composition thus determined shall be reported to the purchaser or his representative, and shall conform as specified in the purchase order.

Check Analyses

8. Analyses may be made by the purchaser from samples representing the bolting material. The chemical composition thus determined shall conform to the requirements specified.

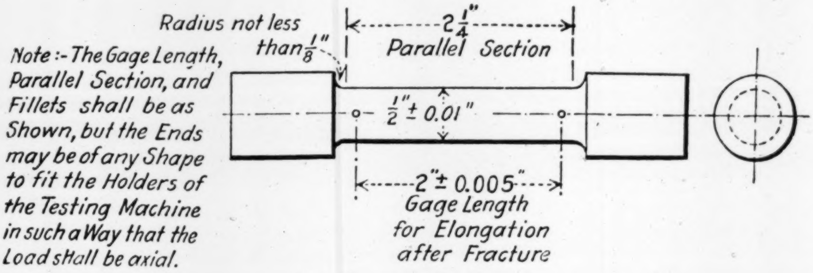
Radius not less than $\frac{1}{8}$"
$2\frac{1}{4}$"
Parallel Section
$\frac{1}{2}$" ± 0.01"
2" ± 0.005"
Gage Length for Elongation after Fracture
Note :- The Gage Length, Parallel Section, and Fillets shall be as Shown, but the Ends may be of any Shape to fit the Holders of the Testing Machine in such a Way that the Load shall be axial.

FIG. 1.—Standard 2-in. Gage Length Tension Test Specimen.

Physical Properties

9. (*a*) Materials shall conform to the following requirements as to minimum tensile properties at room temperature after oil quenching and drawing as noted in Table I.

(*b*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine, or by the use of dividers or other approved method, at a crosshead speed not to exceed $\frac{1}{8}$ in. per min. Where a definite yield point is not exhibited, the yield strength corresponding to a limiting permanent set of 0.2 per cent of the gage length of the specimen, shall be reported instead. The "offset method" of determining yield strength as described in the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials[1] shall be followed. The tensile strength shall be determined at a crosshead speed not to exceed $1\frac{1}{2}$ in. per min.

Test Specimens

10. (*a*) Tension test specimens taken from bar stock, finished bolts screws, or studs shall conform to the dimensions shown in Fig. 1.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 833.

TABLE I.—TENSILE REQUIREMENTS FOR ALLOY-STEEL BOLTING.

Grade	Diameter, In.	Minimum Draw Temperature, deg. Fahr.	Tensile Strength, min., lb. per sq. in.	Yield Point, min., lb. per sq. in.	Elongation in 2 in., min., per cent	Reduction of Area, min., per cent
FERRITIC STEELS						
B4 Nickel-Chromium-Molybdenum	$2\frac{1}{2}$ and under	1000	160 000	135 000	14	45
		1100	140 000	120 000	15	50
		1200	125 000	105 000	16	50
	Over $2\frac{1}{2}$ to 4, incl.	1000	155 000	130 000	14	45
		1100	135 000	115 000	15	50
		1200	120 000	100 000	16	50
B5 4 to 6 per cent Chromium	$2\frac{1}{2}$ and under	1000	120 000	95 000	15	45
		1100	110 000	90 000	16	50
		1200	100 000	85 000	17	55
	Over $2\frac{1}{2}$ to 4, incl.	1000	105 000	85 000	15	45
		1100	100 000	80 000	16	50
		1200	95 000	75 000	17	55
B6 13 per cent Chromium	$2\frac{1}{2}$ and under	1000	150 000	125 000	13	45
		1100	120 000	100 000	15	50
		1200	105 000	85 000	17	55
	Over $2\frac{1}{2}$ to 4, incl.	1000	140 000	120 000	13	45
		1100	115 000	95 000	15	50
		1200	100 000	80 000	17	55
B7 Chromium-Molybdenum	$2\frac{1}{2}$ and under	1000	135 000	115 000	15	50
		1100	125 000	105 000	16	50
		1200	105 000	90 000	17	55
	Over $2\frac{1}{2}$ to 4, incl.	1000	125 000	105 000	15	50
		1100	115 000	95 000	16	55
		1200	105 000	85 000	17	55
B7a Chromium-High-Molybdenum	$2\frac{1}{2}$ and under	1000	135 000	115 000	15	50
		1100	125 000	105 000	16	50
		1200	105 000	90 000	17	55
	Over $2\frac{1}{2}$ to 4, incl.	1000	125 000	105 000	15	50
		1100	115 000	95 000	16	55
		1200	105 000	85 000	17	55
B11 Tungsten-Chromium-Vanadium	$2\frac{1}{2}$ and under	1000	210 000	190 000	11	35
		1100	205 000	180 000	12	35
		1200	185 000	165 000	13	40
	Over $2\frac{1}{2}$ to 4, incl.	1000	205 000	185 000	10	35
		1100	200 000	175 000	11	35
		1200	180 000	160 000	12	40
B12 Nickel-Chromium	$2\frac{1}{2}$ and under	1000	125 000	105 000	16	50
		1100	115 000	95 000	17	50
		1200	105 000	85 000	18	55
	Over $2\frac{1}{2}$ to 4, incl.	1000	120 000	100 000	16	50
		1100	110 000	90 000	17	50
		1200	100 000	80 000	18	55
B13 Tungsten-Molybdenum-Chromium	$2\frac{1}{2}$ and under	1000	155 000	130 000	14	45
		1100	135 000	115 000	15	50
		1200	120 000	100 000	16	55
	Over $2\frac{1}{2}$ to 4, incl.	1000	145 000	120 000	14	45
		1100	125 000	105 000	15	50
		1200	115 000	95 000	16	55
B14[a] Chromium-Molybdenum-Vanadium	$2\frac{1}{2}$ and under	1000	145 000	120 000	14	45
		1100	135 000	115 000	15	45
		1200	125 000	105 000	16	50
	Over $2\frac{1}{2}$ to 4, incl.	1000	......	......	....	....
		1100	......	......	....	....
		1200	......	......	....	....
B15 Silicon-Chromium-Molybdenum	$2\frac{1}{2}$ and under	1000	160 000	135 000	14	45
		1100	140 000	120 000	15	50
		1200	125 000	105 000	16	50
	Over $2\frac{1}{2}$ to 4, incl.	1000	155 000	130 000	14	45
		1100	135 000	115 000	15	45
		1200	120 000	100 000	16	50
AUSTENITIC STEEL						
B8 18 Chromium 8 Nickel	All diameters	2000 F., water quench	75 000	30 000	35	50

[a] Special normalizing treatment required.

(*b*) In the case of small sections which will not permit of taking the standard tension test specimen specified in Paragraph (*a*), the tension test specimen shall be as large as feasible and its dimensions shall be proportional to those shown in Fig. 1. The gage length for measuring elongation shall be four times the diameter of the specimen.

(*c*) Specimens for tension tests shall be taken longitudinally. For sizes under 1½ in. in diameter, the full section shall be turned to conform to requirements specified in Paragraph (*a*). For sizes 1½ in. and larger, the specimen shall be taken half-way between the center and surface.

Number of Tests

11. (*a*) For bars, one tension test shall be made from each tempering charge. If more than one quenching charge is represented in a tempering charge, one tension test shall be made from each quenching charge. If more than one melt is represented in a quenching charge, one tension test shall be made from each melt.

(*b*) Except as specified in Paragraph (*c*), one tension test shall be made for each lot of 300 pieces or fraction thereof of bolts, screws and studs for sizes up to and including 2½ in. in diameter; or for each lot of 100 pieces or fraction thereof for sizes over 2½ in.

(*c*) Unless required on the order, tension tests shall not be made on an order of less quantity than specified in Paragraph (*b*); in which case acceptance shall be based upon the provisions in Section 2 or 3.

(*d*) If any test specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

(*e*) If the percentage of elongation of any tension test specimen is less than that specified in Section 10 (*a*) and any part of the fracture is more than ¾ in. from the center of the gage length, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

Retest

12. If the results of the physical tests of any test lot do not conform to the requirements specified, the manufacturer may retreat such lot not more than twice, in which case two additional tension tests shall be made from such lot, all of which shall conform to the requirements specified.

Threads

13. (*a*) The threads on bolts, screws, and studs shall be in accordance with the American Standard for Screw Threads. (A.S.A. No.: B1. 1-1935.)

(*b*) Where practical, all threads shall be formed after heat treatment.

Finish

14. Bolts, screws, and studs shall be pointed and shall have a workmanlike finish.

Marking

15. The serial marking for identification of material and the manufacturer's identification mark shall be stamped on the top of the head of bolts and screws, and on one end of studs. The identification symbol shall be as shown in Section 6.

Inspection

16. The inspector representing the purchaser shall have free entry at all times, while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the bolts, screws and studs ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the bolts, screws, and studs are being furnished in accordance with these specifications. All tests (except check analysis) and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection

17. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 9 shall be reported within five working days from the receipt of samples.

(*b*) Bolts, screws, or studs which show injurious defects subsequent to their acceptance at the manufacturer's works will be rejected and the manufacturer will be notified.

Rehearing

18. Samples tested in accordance with Section 9 which represent rejected bolts, screws or studs shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the test, the manufacturer may make claim for a hearing within that time.

EXPLANATORY NOTES

NOTE 1.—The subcommittee formulating these specifications has included several grades of material that have been rather extensively used. Upon agreement between the purchaser and the manufacturer, other grades of material may be employed, provided they otherwise meet the requirements of these specifications. Other compositions will be considered for inclusion in the specifications by the committee from time to time as the need becomes apparent.

NOTE 2.—High-temperature properties, when it is desired that they be indicated or specified, shall be a matter of agreement between the purchaser and the manufacturer. No high-temperature tests shall be required on the materials selected unless made a matter of agreement between the purchaser and the manufacturer.

TENTATIVE SPECIFICATIONS
FOR
SEAMLESS ALLOY-STEEL PIPE FOR SERVICE AT TEMPERATURES FROM 750 TO 1100 F.[1]

A.S.T.M. Designation: A 158 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1934; ADOPTED IN AMENDED FORM, 1936; REISSUED AS TENTATIVE, 1937.[2]

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

Scope

1. (*a*) These specifications are intended as a general guide for the purchase of alloy-steel pipe and to serve as a basis for agreement between the purchaser and the manufacturer as to details not specifically covered.

(*b*) These specifications cover seamless alloy-steel pipe intended for service at metal temperatures from 750 to 1100 F.; both ferritic and austenitic steels, designated P 3a, P 6, etc., are included (see Explanatory Note 1). The maximum temperature at which the pipe will be operated in service shall be stated in the purchase order. Choice from the respective steels should be made on the basis of the requirements of design, service conditions, and the physical properties (see Explanatory Note 2).

(*c*) Supplementary requirements (Paragraphs S1 to S6) of an optional nature are provided for pipe intended for use in central stations having steam service pressures of 400 lb. per sq. in. or over and temperatures up to 1100 F., or other applications where a superior grade of pipe is required. These supplementary requirements call for additional tests to be made at the expense of the purchaser, and when desired shall be so stated in the order.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-1 on Steel.

These specifications, together with the Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A.S.T.M. Designation: A 206 - 37 T), see p. 633, are in effect a revision of, and replace the former Standard Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A.S.T.M. Designation: A 158 - 36), which standard was accordingly discontinued in 1937.

[2] Accepted for publication as tentative by Committee E-10 on Standards, August 26, 1937.

Certification

2. Where mutually agreed upon in writing between the manufacturer and the purchaser, a certification that the material conforms to the requirements of these specifications shall be the basis of acceptance of the material. Otherwise, the manufacturer shall report to the purchaser or his representative the results of the chemical analyses and physical tests made in accordance with these specifications.

Process

3. (*a*) The steel shall be made by either or both of the following processes: open-hearth or electric-furnace.

(*b*) Unless otherwise specified, all pipe 2 in. and over in nominal diameter shall be furnished hot-finished followed by the treatment specified in Section 5.

(*c*) Unless otherwise specified, pipe under 2 in. in nominal diameter may be furnished either hot-finished or cold-drawn, with, where necessary, a suitable manufacturer's finishing treatment.

Discard

4. Sufficient discard shall be made from each ingot to secure freedom from injurious piping and undue segregation.

Heat Treatment

5. (*a*) All ferritic steels covered in Section 3 other than Grade P 5c shall be reheated and furnished in a full-annealed or normalized and drawn condition as stated in the purchase order. If furnished in the normalized and drawn condition, the temperature for drawing shall be at least 200 F. above the service temperature as given in the purchase order (Note 1).

(*b*) Grade P 5c steel, unless otherwise specified, shall be treated by heating to approximately 1350 F. for a proper time, followed by air or furnace cooling.

(*c*) All austenitic steels shall be heat treated (Note 2) according to present accepted practice.

NOTE 1.—Certain of the ferritic steels covered by these specifications will harden if cooled rapidly from above their critical temperature. Some will air harden, that is, become hardened to an undesirable degree when cooled in air from high temperatures, particularly the 4 to 6 per cent chromium steels, P 5. Therefore, operations involving heating such steels above their critical temperatures, such as welding, flanging and hot bending, should be followed by suitable heat treatment.

NOTE 2.—Fabricating operations involving upsetting, flanging, bending, and forming often make retreating desirable to insure adequate corrosion resistance, depending upon service conditions.

Chemical Composition[1]

6. Each alloy[2] shall conform to the following requirements as to chemical composition,[3] or to other chemical composition requirements as specified in the purchase order:

Class	Ferritic Steels					
Identification Symbol	P 3a	P 3b	P 5a	P 5b	P 5c	P 6
Grade	Chromium-Molybdenum	Chromium-Molybdenum	4 to 6 per cent Chromium	4 to 6 per cent Chromium-Silicon-Molybdenum	4 to 6 per cent Chromium-Molybdenum Stabilized with Titanium or Columbium	13 per cent Chromium
Carbon, per cent	0.15 max.	0.15 max.	0.15 max. or 0.20 max.	0.15 max.	As specified 0.10 or 0.15 max.	0.12 max.
Manganese, per cent	0.40 to 0.60	0.30 to 0.60	0.50 max.	0.30 max.	0.50 max.	0.50 max.
Phosphorus, max., per cent	0.04	0.03	0.03	0.03	0.03	0.03
Sulfur, max., per cent	0.05	0.03	0.03	0.03	0.03	0.03
Silicon, per cent	0.45 to 0.75	0.50 max.	0.50 max.	1.00 to 2.00	0.50 max.	0.50 max.
Nickel, per cent	....	....	....	....	....	....
Chromium, per cent	1.50 to 2.00	1.75 to 2.25	4.00 to 6.00	4.00 to 6.00	4.00 to 6.00	12.00 to 15.00
Molybdenum, per cent	0.60 to 0.80	0.45 to 0.65	0.45 to 0.65[a]	0.45 to 0.65	0.45 to 0.65	....
Tungsten, per cent	....	....	0.75 to 1.25[a]	....	....	2.50 to 3.50
Titanium	....	....	....	....	4 to 6 times carbon	....
or Columbium	....	....	....	....	8 to 10 times carbon	....

Class	Ferritic Steels		Austenitic Steels		
Identification Symbol	P 11	P 15	P 8a	P 8b	P 8c[b]
Grade	Chromium-Molybdenum	Silicon-Molybdenum	18 Chromium-8 Nickel	18 Chromium 10 Nickel Stabilized with Titanium or Columbium	18 Chromium 8 Nickel 2 Silicon
Carbon, max., per cent	0.15	0.15	0.07	0.08	0.25
Manganese, per cent	0.30 to 0.60	0.30 max.	0.20 to 0.70	0.70 max.	0.50 to 0.70
Phosphorus, per cent	0.04	0.04	0.03	0.03	0.03
Sulfur, per cent	0.05	0.045	0.03	0.03	0.03
Silicon, per cent	0.50 to 1.00	1.15 to 1.65	0.75 max.	0.50 max.	1.00 to 3.00
Nickel, per cent	....	....	8.00 to 10.00	9.00 to 12.00	8.00 to 10.00
Chromium, per cent	1.00 to 1.50	....	17.00 to 20.00	17.00 to 19.00	17.00 to 19.00
Molybdenum, per cent	0.45 to 0.65	0.45 to 0.65	....	....	....
Tungsten, per cent	....	....	....	....	....
Titanium	....	....	....	0.35 min.	....
or Columbium	....	....	....	8 times carbon content, min.	....

[a] Either molybdenum or tungsten shall be used.
[b] Grade P8c was formerly designated P13.

Ladle Analysis

7. A ladle analysis of each melt of steel shall be made by the steel manufacturer. The chemical composition thus determined, or that

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[1] When material is ordered for welding fabrication, chemical and physical requirements shall be a matter of agreement between the purchaser and the manufacturer.

[2] Alloys have been numbered so that similar alloys in the corresponding specifications for forgings, castings, and bolting will bear the same number for alloys numbered 10 or below. No alloys P 4, P 7 or P 10 are included herein as the particular compositions corresponding to these numbers are not included in these specifications.

[3] The list is incomplete and will be reviewed annually and revised promptly when necessary.

determined from a check analysis made by the pipe manufacturer, if the latter has not manufactured the steel, shall be reported to the purchaser or his representative and shall conform to the requirements specified in Section 6.

Check Analysis

8. (*a*) At the request of the purchaser's inspector, analyses as specified below shall be made by the manufacturer from the finished pipe and shall conform to the requirements specified in Section 6:

NOMINAL DIAMETER	NUMBER OF PIPES SELECTED
Under 2 in.	2 pipes from each lot of 400 lengths or fraction thereof
2 to 5 in. inclusive	2 pipes from each lot of 200 lengths or fraction thereof
6 in. and over	2 pipes from each lot of 100 lengths or fraction thereof

Results of these analyses shall be reported to the purchaser or his representative and shall conform to the requirements specified in Section 6.

(*b*) Drillings for analysis from pipe shall be taken from several points around each pipe selected for analysis. Drillings for analysis from billets shall be taken at a point midway between the outside and center of the billet by drilling parallel to the axis.

(*c*) If the analysis of one of the tests specified in Section 7 or 8 (*a*) does not conform to the requirements specified in Section 6, analysis of each billet or pipe from the same heat or lot may be made, and all billets or pipe conforming to the requirements shall be accepted.

Tension Tests

9. (*a*) The material shall conform to the following minimum requirements as to tensile properties[1] at room temperature:

	FERRITIC STEELS	AUSTENITIC STEELS
Tensile strength, min., lb. per sq. in.	60 000[a]	75 000[a]
Yield point, min., lb. per sq. in.:		
Fully annealed	25 000[a]	......
Normalized and drawn	30 000[a]	......
Rapidly air-cooled or quenched	......	30 000[a]
Elongation in 2 in., min., per cent	30[a]	50[a]

[a] For transverse tension tests the material shall conform to these minimum tensile requirements except that the elongation in 2 in. shall be 25 per cent for the ferritic steels and 40 per cent for the austenitic steels.

(*b*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine, or by the use of dividers or other approved method, at a cross head speed not to exceed $\frac{1}{8}$ in. per min. Where a definite yield point is not exhibited, the yield strength, corresponding to a limiting permanent set of 0.2 per cent of the gage length of the specimen, shall be reported instead. The "offset method" of determining yield strength as described in the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials[1] shall be followed. The tensile strength shall be determined at a crosshead speed not to exceed $1\frac{1}{2}$ in. per minute.

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[1] When material is ordered for welding fabrication, chemical and physical requirements shall be matter of agreement between the purchaser and the manufacturer.

Hydrostatic Tests

10. (*a*) Unless otherwise mutually agreed upon between the manufacturer and the purchaser, each length of pipe shall be tested by the manufacturer to a hydrostatic pressure which will produce in the pipe wall a stress of 50 per cent of the minimum specified yield point at room temperature. This pressure shall be determined by the formula:

$$P = \frac{2St}{D}$$

where P = the minimum hydrostatic test pressure in pounds per square inch,

S = 0.50 times the minimum specified yield point at room temperature in pounds per square inch,

t = the nominal wall thickness in inches, and

D = the outside diameter in inches.

(*b*) The maximum hydrostatic test pressure shall not exceed 2500 lb. per sq. in. for sizes 3 in. or under in outside diameter, nor 4500 lb. per sq. in. for sizes over 3 in. to but not including $8\frac{5}{8}$ in. in outside diameter, and 3000 lb. per sq. in. for all sizes $8\frac{5}{8}$ in. or over in outside diameter. The hydrostatic pressure shall be maintained for not less than 5 sec.

Flattening Tests

11. For pipe over 2 in. in nominal diameter, a section of pipe not less than $2\frac{1}{2}$ in. in length shall be flattened between parallel plates until the opposite walls of the pipe meet. No cracks or breaks in the metal shall occur until the distance between the plates is less than one half the outside diameter of the pipe, but in no case less than seven times the thickness of the pipe wall. Evidence of laminations or burnt material shall not develop during the entire flattening process.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 833.

Bend Tests

12. For pipe 2 in. or under in nominal diameter, a sufficient length of pipe shall stand being bent cold through 90 deg. around a cylindrical mandrel, the diameter of which is twelve times the nominal diameter of the pipe, without developing cracks. When ordered for close coiling, the pipe shall stand being bent cold through 180 deg. around a cylindrical mandrel, the diameter of which is eight times the nominal diameter of the pipe, without failure.

Test Specimens

13. (*a*) Specimens cut either longitudinally or transversely shall be acceptable for tension tests.

(*b*) Longitudinal tension tests may be made in full section of the pipe up to the capacity of the testing machine. For larger sizes, the tension test specimens shall consist of strips cut from the pipe. The width of these specimens shall be 1½ in. and shall have a gage length of 2 in. When the pipe wall thickness exceeds ¾ in., the A.S.T.M. standard 0.505-in. round specimen shall be used (Note). Longitudinal tension specimens shall not be flattened between gage marks. The sides of specimens shall be parallel between gage marks.

(*c*) The transverse tension test may be made on pipe 8 in. and over in nominal diameter. Specimens may be taken from a ring cut from the pipe or from sections resulting from the flattening tests. The specimen shall consist of a strip cut transversely from the pipe; the width of the specimen shall be 1½ in. and its gage length 2 in. When the pipe wall thickness exceeds ¾ in., the A.S.T.M. standard 0.505-in. round specimen shall be used (Note). Specimens shall be flattened hot and heat treated in the same manner as the pipe, and shall be parallel between gage marks. At the option of the manufacturer, the transverse tension test specimen may be machined off on either surface provided not over 15 per cent of the nominal thickness is removed from either side.

NOTE.—The standard tension test specimens are described in the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials.[1]

(*d*) Test specimens for flattening and bend tests shall consist of sections cut from a pipe. Specimens for flattening tests shall be smooth on the ends and free from burrs, except when made on crop ends.

(*e*) All routine check tests shall be made at room temperature.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 833.

Number of Tests

14. (*a*) Tests shall be made as follows on one pipe from each heat-treated lot, but in no case on less than 5 per cent of the pipe ordered. Results of these tests shall be reported to the purchaser or his representative:

One transverse or longitudinal tension test specified in Section 9.

The flattening test specified in Section 11 for pipe over 2 in. in nominal diameter.

The bend test specified in Section 12 for pipe 2 in. or under in nominal diameter.

(*b*) For material heat treated by the continuous process, the tests specified in Paragraph (*a*) shall be made on each pipe in a lot constituting 5 per cent of the pipe ordered, but on not less than two pipes.

(*c*) Each length of pipe shall be subjected to the hydrostatic test specified in Section 10.

Retests

15. (*a*) If the results of any physical tests of any lot do not conform to the requirements specified in Sections 9, 11, or 12, retests shall be made on additional pipes of double the original number from the same lot, each of which shall conform to the requirements specified.

(*b*) If the percentage of elongation of any tension test specimen is less than that specified and any part of the fracture is more than ¾ in. from the center of the gage length of the specimen, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed. If a specimen breaks in an inside or outside surface flaw, a retest shall be allowed.

(*c*) If any specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

Retreatment

16. If individual lengths of pipe selected to represent any lot fail to meet the test requirements specified in Sections 9, 11, or 12, the lot may be heat treated again and resubmitted for test, except that any individual lengths which meet the test requirements before retreating will be acceptable.

Permissible Variations in Weight and Dimensions

17. (*a*) *Weight.*—The weight of any length of pipe in sizes up to and including 12 in. shall not vary more than 3.5 per cent under and 6.5 per cent over that specified. For sizes over 12 in. the weight of any length shall not vary more than 5 per cent under and 10 per

cent over that specified. Unless otherwise mutually agreed upon between the manufacturer and the purchaser, pipe in sizes smaller than 4 in. may be weighed in convenient lots; pipe in sizes 4 in. and larger shall be weighed separately.

(*b*) *Diameter.*—Variations in outside diameter shall not exceed those shown in the following table:

	Permissible Variations in Outside Diameters	
Nominal Diameter	Under	Over
$\frac{1}{8}$ to $1\frac{1}{2}$ in., inclusive	$\frac{1}{32}$ in. (0.031 in.)	$\frac{1}{64}$ in. (0.015 in.)
2 to 4 in., inclusive	$\frac{1}{32}$ in. (0.031 in.)	$\frac{1}{32}$ in. (0.031 in.)
5 to 8 in., inclusive	$\frac{1}{32}$ in. (0.031 in.)	$\frac{1}{16}$ in. (0.062 in.)
10 to 18 in., inclusive	$\frac{1}{32}$ in. (0.031 in.)	$\frac{3}{32}$ in. (0.093 in.)
20 to 24 in., inclusive	$\frac{1}{32}$ in. (0.031 in.)	$\frac{1}{8}$ in. (0.125 in.)

(*c*) *Thickness.*—The minimum wall thickness at any point shall not be more than 12.5 per cent under the nominal wall thickness specified.

(*d*) *Length.*—Pipe lengths shall be in accordance with the following regular practice:

> The lengths required shall be specified in the order. No pipe shall be under the specified length and not more than $\frac{1}{4}$ in. over that specified.
>
> No jointers are permitted unless otherwise specified.

Ends

18. Unless otherwise specified, pipe shall be furnished with plain ends. All burrs at the ends of the pipe shall be removed.

Finish

19. (*a*) The finished pipe shall be reasonably straight and free from injurious defects, and shall have a workmanlike finish.

Depth of Injurious Defects

(*b*) When the depth of defect in the pipe encroaches on the minimum wall thickness (87.5 per cent of the nominal thickness), or is in excess of 12.5 per cent of the nominal wall thickness, such defects shall be considered injurious.

Machining or Grinding Defects

(*c*) Pipe showing moderate slivers may be machined or ground inside or outside to a depth which shall insure the removal of all included scale and slivers, providing the wall thickness is not reduced below the minimum wall thickness required in these specifications. Machining or grinding shall follow inspection of the pipe as rolled, and shall be followed by supplementary visual inspection.

Repair by Welding

(*d*) Repair of injurious defects shall be permitted only subject to the approval of the purchaser and with the further understanding that the composition of the welding rod shall be suitable for the composition of the metal being welded. Welding of injurious defects in no case shall be permitted when the depth of defect exceeds 33⅓ per cent of the nominal pipe wall thickness or the length of repair exceeds 25 per cent of the nominal diameter of the pipe. Defects shall be thoroughly chipped out before welding, and then heat treated in accordance with Section 5. Each length of repaired pipe shall be retested hydrostatically in accordance with Section 10.

Marking

20. Each length of pipe manufactured in accordance with these specifications shall be legibly marked, either by stenciling, stamping, or rolling, with the manufacturer's private identifying mark, together with the symbols A 158 and the alloy symbol (as A 158 – P6) and an additional S if pipe meets the supplementary requirements specified in Paragraphs S1 to S7. Marking shall be within 12 in. of one end of each length. On pipe sizes 4 in. and larger, the weight shall be given. On small diameter pipe which is bundled, the above information may be legibly stamped on a metal tag securely attached to each bundle. When pipe marked as specified in this section is rejected, the designation A 158 shall be canceled.

Inspection

21. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the pipe ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the pipe is being furnished in accordance with these specifications. All tests and inspection shall be made at the place of manufacture prior to shipment and at the manufacturer's expense, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection

22. Each length of pipe which develops injurious defects in shop working or application shall be rejected, and the manufacturer shall be notified. No rejections under these or any other specifications, shall be marked as specified in Section 20 for sale under these specifications except where such pipe fails to comply with the weight requirements alone, in which case it may be sold under the weight specifications with which it does comply.

SUPPLEMENTARY REQUIREMENTS FOR SEAMLESS PIPE FOR USE IN CENTRAL STATIONS AT PRESSURES OF 400 LB. PER SQ. IN., OR OVER, AND TEMPERATURES OF 750 F. TO 1100 F., OR OTHER APPLICATIONS WHERE A SUPERIOR GRADE OF PIPE IS REQUIRED

S1. These requirements shall not be considered unless specified in the order, in which event the supplementary tests shall be made at the mill, unless otherwise agreed upon, at the purchaser's expense and witnessed by his inspector before shipment of the material.

Check Analysis

S2. Check analysis may be made on any length of pipe. Individual lengths failing to meet the chemical requirements specified in Section 6 shall be rejected.

Transverse Tension Tests

S3. Transverse tension tests may be made on specimens from both ends of each length of pipe. If the specimen from either end of any length fails to meet the physical properties specified in Section 9, that length shall be rejected.

Flattening Tests

S4. The flattening tests specified in Section 11 may be made on specimens from both ends of each length of pipe. Crop ends may be used. If the specimen from either end of any length fails to meet the specified requirements, that length shall be rejected.

Finish and Metal Structure

S5. The finished pipe shall be free from all immoderate slivers and depressions. The steel shall have a homogeneous structure as shown by the etching test in Paragraph S6.

Etching Tests

S6. Etching tests[1] may be made on sections from any pipe, and shall show sound and reasonably uniform material, free from injurious laminations, cracks and similar objectionable defects. Sections used for the flattening tests described in Section 11 may be used for the etching test. If the specimen from any length shows objectionable defects, that length may be rejected.

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[1] Pending development of etching methods applicable to the product covered by these specifications, it is recommended the Tentative Recommended Practice for a Macro-Etch Test for Steel described in the *National Metals Handbook*, Am. Soc. for Metals, 1936 edition, p. 573, be followed.

Rejection

S7. At the purchaser's discretion, if 10 per cent of the pipe in any one lot fail to conform to the requirements of Paragraphs S2 to S6, the entire lot may be rejected. Rejection of a lot shall be based on tests from not less than two pipes. A lot shall consist of all the pipe bought by a purchaser of the same size and wall thickness from any one heat.

EXPLANATORY NOTES

NOTE 1.—The subcommittee formulating these specifications has included several classes of material that have been rather extensively used. Upon mutual agreement between the manufacturer and the purchaser, other classes of material may be employed, provided they otherwise meet the requirements of these specifications. Other compositions will be considered for inclusion in these specifications by the committee from time to time as the need becomes apparent.

NOTE 2.—Pending the incorporation of high-temperature data in an appendix, which it is the purpose in due time to add, high-temperature properties, when it is desired that the same be indicated or specified, shall be a matter of agreement between the manufacturer and the purchaser. No high-temperature tests shall be required on the materials selected unless a matter of agreement between the manufacturer and the purchaser. When required, the short-time tests at elevated temperature shall be in accordance with the Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (A.S.T.M. Designation: E 21 – 37 T)[1] and the creep tests shall be in accordance with the Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (A.S.T.M. Designation: E 22 – 35 T)[2] of the American Society for Testing Materials.

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[1] See p. 1290.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I p. 1291 (1935); also 1937 Book of A.S.T.M. Tentative Standards, p. 1544.

TENTATIVE SPECIFICATIONS
FOR
SEAMLESS CARBON-MOLYBDENUM ALLOY-STEEL PIPE FOR SERVICE AT TEMPERATURES FROM 750 TO 1000 F.[1]

A.S.T.M. Designation: A 206 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.[2]

Scope

1. (*a*) These specifications cover seamless carbon-molybdenum alloy-steel pipe intended for service at metal temperatures from 750 to 1000 F. The maximum temperature at which the pipe will be operated in service shall be stated in the purchase order (see Explanatory Note). Pipe ordered under these specifications shall be suitable for bending, flanging (vanstoning), and similar forming operations, and for fusion welding.

(*b*) Supplementary requirements (Paragraphs S1 to S6) of an optional nature are provided for pipe intended for use in central stations having steam service pressures of 400 lb. per sq. in. or over and temperatures up to 1000 F., or other applications where a superior grade of pipe is required. These supplementary requirements call for additional tests to be made at the expense of the purchaser, and when desired shall be so stated in the order.

Certification

2. Where mutually agreed upon in writing between the manufacturer and the purchaser, a certification that the material conforms to the requirements of these specifications shall be the basis of acceptance of the material. Otherwise, the manufacturer shall report to the purchaser or his representative the results of the chemical analyses and physical tests made in accordance with these specifications.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-1 on Steel.

These specifications, together with the Tentative Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A.S.T.M. Designation: A 158 – 37 T), see p. 622, are in effect a revision of, and replace the former Standard Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A.S.T.M. Designation: A 158 – 36), which standard was accordingly discontinued in 1937.

[2] Accepted for publication as tentative by Committee E-10 on Standards, August 26, 1937.

Process

3. (*a*) The steel shall be made by either or both of the following processes: open-hearth or electric-furnace.

(*b*) Unless otherwise specified, all pipe 2 in. and over in nominal diameter shall be furnished hot-finished followed by the treatment specified in Section 5.

(*c*) Unless otherwise specified, pipe under 2 in. in nominal diameter may be furnished either hot-finished or cold-drawn, with, where necessary, a suitable manufacturer's finishing treatment.

Discard

4. Sufficient discard shall be made from each ingot to secure freedom from injurious piping and undue segregation.

Heat Treatment

5. The finished pipe shall be furnished in the normalized and drawn condition unless otherwise specified in the purchase order. Normalizing shall consist of reheating the finished pipe to 1650 F. and cooling in still air to 800 F. or below. Drawing shall consist of reheating the normalized pipe to 1200 F. and allowing to cool slowly in still air.

Chemical Composition

6. The steel shall conform to the following requirements as to chemical composition:

Identification Symbol[a]	P 1
Grade	Carbon-Molybdenum
Carbon, per cent	0.10 to 0.20
Manganese, per cent	0.30 to 0.60
Phosphorus, max., per cent	0.04
Sulfur, max., per cent	0.05
Silicon, per cent	0.10 to 0.50
Molybdenum, per cent	0.45 to 0.65

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[a] The similar alloy in the corresponding specifications for alloy-steel forgings and castings bears the number F 1 and C 1, respectively.

Ladle Analysis

7. A ladle analysis of each melt of steel shall be made by the steel manufacturer. The chemical composition thus determined, or that determined from a check analysis made by the pipe manufacturer, if the latter has not manufactured the steel, shall be reported to the purchaser or his representative and shall conform to the requirements specified in Section 6.

Check Analysis

8. (*a*) At the request of the purchaser's inspector, analyses as specified below shall be made by the manufacturer from the finished pipe and shall conform to the requirements specified in Section 6:

NOMINAL DIAMETER	NUMBER OF PIPE SELECTED
Under 2 in.	2 pipes from each lot of 400 lengths or fraction thereof
2 to 5 in., inclusive	2 pipes from each lot of 200 lengths or fraction thereof
6 in. and over	2 pipes from each lot of 100 lengths or fraction thereof

Results of these analyses shall be reported to the purchaser or his representative and shall conform to the requirements specified in Section 6.

(*b*) Drilling for analysis from pipe shall be taken from several points around each pipe selected for analysis. Drillings for analysis from billets shall be taken at a point midway between the outside and center of the billet by drilling parallel to the axis.

(*c*) If the analysis of one of the tests specified in Section 7 or Paragraph (*a*) of this section does not conform to the requirements specified in Section 6, analysis of each billet or pipe from the same heat or lot may be made, and all billets or pipe conforming to the requirements shall be accepted.

Tension Tests

9. (*a*) The material shall conform to the following minimum requirements as to tensile properties[a] at room temperature:

Tensile strength, min., lb. per sq. in.	55 000
Yield point, min., lb. per sq. in.	30 000
Elongation in 2 in., min., per cent	30

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[a] For transverse tension tests the material shall conform to these minimum tensile requirements except that the elongation in 2 in. shall be 25 per cent.

(*b*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine, or by the use of dividers or other approved method, at a crosshead speed not to exceed $\frac{1}{8}$ in. per min. Where a definite yield point is not exhibited, the yield strength, corresponding to a limiting permanent set of 0.2 per cent of the gage length of the specimen, shall be reported instead. The "offset method" of determining yield strength as described in the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials[1] shall be followed. The tensile strength shall be determined at a crosshead speed not to exceed $1\frac{1}{2}$ in. per min.

Hydrostatic Tests

10. (*a*) Unless otherwise mutually agreed upon between the manufacturer and the purchaser, each length of pipe shall be tested by the manufacturer to a hydrostatic pressure which will produce in the pipe wall a stress of 50 per cent of the minimum specified yield point at room temperature. This pressure shall be determined by the formula:

$$P = \frac{2St}{D}$$

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 833.

where P = the minimum hydrostatic test pressure in pounds per square inch,
S = 0.50 times the minimum specified yield point at room temperature in pounds per square inch,
t = the nominal wall thickness in inches, and
D = the outside diameter in inches.

(*b*) The maximum hydrostatic test pressure shall not exceed 2500 lb. per sq. in. for sizes 3 in. or under in outside diameter, nor 4500 lb. per sq. in. for sizes over 3 in. to but not including 8⅝ in. in outside diameter and 3000 lb. per sq. in. for all sizes 8⅝ in. or over in outside diameter. The hydrostatic pressure shall be maintained for not less than 5 sec.

Flattening Tests

11. For pipe over 2 in. in nominal diameter, a section of pipe not less than 2½ in. in length shall be flattened between parallel plates until the opposite walls of the pipe meet. No cracks or breaks in the metal shall occur until the distance between the plates is less than one-half the outside diameter of the pipe, but in no case less than seven times the thickness of the pipe wall. Evidence of laminations or burnt material shall not develop during the entire flattening process.

Bend Tests

12. For pipe 2 in. or under in nominal diameter, a sufficient length of pipe shall stand being bent cold through 90 deg. around a cylindrical mandrel, the diameter of which is twelve times the nominal diameter of the pipe, without developing cracks. When ordered for close coiling, the pipe shall stand being bent cold through 180 deg. around a cylindrical mandrel, the diameter of which is eight times the nominal diameter of the pipe, without failure.

Test Specimens

13. (*a*) Specimens cut either longitudinally or transversely shall be acceptable for tension tests.

(*b*) Longitudinal tension tests may be made in full section of the pipe up to the capacity of the testing machine. For larger sizes, the tension test specimens shall consist of strips cut from the pipe. The width of these specimens shall be 1½ in. and shall have a gage length of 2 in. When the pipe wall thickness exceeds ¾ in., the A.S.T.M. standard 0.505-in. round specimen shall be used (Note). Longitudinal tension specimens shall not be flattened between gage marks. The sides of specimens shall be parallel between gage marks.

(*c*) The transverse tension test may be made on pipe 8 in. and over in nominal diameter. Specimens may be taken from a ring cut from the pipe or from sections resulting from the flattening tests. The specimen shall consist of a strip cut transversely from the pipe; the width of the specimen shall be $1\frac{1}{2}$ in. and its gage length 2 in. When the pipe wall thickness exceeds $\frac{3}{4}$ in. the A.S.T.M. standard 0.505-in. round specimen shall be used (Note). Specimens shall be flattened hot and heat treated in the same manner as the pipe, and shall be parallel between gage marks. At the option of the manufacturer, the transverse tension test specimen may be machined off on either surface provided not over 15 per cent of the nominal thickness is removed from either side.

NOTE.—The standard tension test specimens are described in the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials.[1]

(*d*) Test specimens for flattening and bend tests shall consist of sections cut from a pipe. Specimens for flattening tests shall be smooth on the ends and free from burrs, except whem made on crop ends.

(*e*) All routine check tests shall be made at room temperature.

Number of Tests

14. (*a*) Tests shall be made as follows on one pipe from each heat-treated lot, but in no case on less than 5 per cent of the pipe ordered. Results of these tests shall be reported to the purchaser or his representative:

One transverse or longitudinal tension test specified in Section 9.

The flattening test specified in Section 11 for pipe over 2 in. in nominal diameter.

The bend test specified in Section 12 for pipe 2 in. or under in nominal diameter.

(*b*) For material heat treated by the continuous process, the tests specified in Paragraph (*a*) shall be made on each pipe in a lot constituting 5 per cent of the pipe ordered, but on not less than two pipes.

(*c*) Each length of pipe shall be subjected to the hydrostatic test specified in Section 10.

Retests

15. (*a*) If the results of any physical tests of any lot do not conform to the requirements specified in Sections 9, 11, or 12, retests shall be made on additional pipes of double the original number from the same lot, each of which shall conform to the requirements specified.

(*b*) If the percentage of elongation of any tension test specimen is less than that specified and any part of the fracture is more than $\frac{3}{4}$ in. from the center of the gage length of the specimen, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed. If a specimen breaks in an inside or outside surface flaw, a retest shall be allowed.

(*c*) If any specimen shows defective machining or develops flaws, it may be discarded and another specimen substituted.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 833.

Retreatment

16. If individual lengths of pipe selected to represent any lot fail to meet the test requirements specified in Sections 9, 11, or 12, the lot may be heat treated again and resubmitted for test, except that any individual lengths which meet the test requirements before retreating will be acceptable.

Permissible Variations in Weight and Dimensions

17. (*a*) *Weight.*—The weight of any length of pipe in sizes up to and including 12 in. shall not vary more than 3.5 per cent under and 6.5 per cent over that specified. For sizes over 12 in. the weight of any length shall not vary more than 5 per cent under and 10 per cent over that specified. Unless otherwise mutually agreed upon between the manufacturer and the purchaser, pipe in sizes smaller than 4 in. may be weighed in convenient lots; pipe in sizes 4 in. and larger shall be weighed separately.

(*b*) *Diameter.*—Variations in outside diameter shall not exceed those shown in the following table:

NOMINAL DIAMETER	PERMISSIBLE VARIATIONS IN OUTSIDE DIAMETER	
	UNDER	OVER
⅛ to 1½ in., inclusive	$\frac{1}{32}$ in. (0.031 in.)	$\frac{1}{64}$ in. (0.015 in.)
2 to 4 in., inclusive	$\frac{1}{32}$ in. (0.031 in.)	$\frac{1}{32}$ in. (0.031 in.)
5 to 8 in., inclusive	$\frac{1}{32}$ in. (0.031 in.)	$\frac{1}{16}$ in. (0.062 in.)
10 to 18 in., inclusive	$\frac{1}{32}$ in. (0.031 in.)	$\frac{3}{32}$ in. (0.093 in.)
20 to 24 in., inclusive	$\frac{1}{32}$ in. (0.031 in.)	⅛ in. (0.125 in.)

(*c*) *Thickness.*—The minimum wall thickness at any point shall not be more than 12.5 per cent under the nominal wall thickness specified.

(*d*) *Length.*—Pipe lengths shall be in accordance with the following regular practice:

> The lengths required shall be specified in the order. No pipe shall be under the specified length and not more than ¼ in. over that specified.
>
> No jointers are permitted unless otherwise specified.

Ends

18. Unless otherwise specified, pipe shall be furnished with plain ends. All burrs at the ends of the pipe shall be removed.

Finish

19. (*a*) The finished pipe shall be reasonably straight and free from injurious defects, and shall have a workmanlike finish.

Depth of Injurious Defects

(*b*) When the depth of defect in the pipe encroaches on the minimum wall thickness (87.5 per cent of the nominal thickness), or is in excess of 12.5 per cent of the nominal wall thickness, such defects shall be considered injurious.

Machining or Grinding Defects

(*c*) Pipe showing moderate slivers may be machined or ground inside or outside to a depth which shall insure the removal of all included scale and slivers, providing the wall thickness is not reduced below the minimum wall thickness required in these specifications. Machining or grinding shall follow inspection of the pipe as rolled, and shall be followed by supplementary visual inspection.

Repair by Welding

(*d*) Repair of injurious defects shall be permitted only subject to the approval of the purchaser and with the further understanding that the composition of the welding rod shall be suitable for the composition of the metal being welded. Welding of injurious defects in no case shall be permitted when the depth of defect exceeds 33⅓ per cent of the nominal pipe wall thickness or the length of repair exceeds 25 per cent of the nominal diameter of the pipe. Defects shall be thoroughly chipped out before welding, and then heat treated in accordance with Section 5. Each length of repaired pipe shall be retested hydrostatically in accordance with Section 10.

Marking

20. Each length of pipe manufactured in accordance with these specifications shall be legibly marked, either by stenciling, stamping, or rolling, with the manufacturer's private identifying mark, together with the symbols A 206 and the alloy symbol (as A 206 - P1) and an additional S if pipe meets the supplementary requirements specified in Paragraphs S1 to S7. Marking shall be within 12 in. of one end of each length. On pipe sizes 4 in. and larger, the weight shall be given. On small diameter pipe which is bundled, the above information may be legibly stamped on a metal tag securely attached to each bundle. When pipe marked as specified in this section is rejected, the designation A 206 shall be canceled.

Inspection

21. The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the pipe ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the pipe is being furnished in accordance with these specifications. All tests and inspection shall be made at the place of manufacture prior to shipment and at the manufacturer's expense, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works. The manufacturer shall notify the purchaser in time so that he may have his inspector present to witness any part of the manufacture or tests that may be desired.

Rejection

22. Each length of pipe which develops injurious defects in shop working or application shall be rejected, and the manufacturer shall be notified. No rejections under these or any other specifications, shall be marked as specified in Section 20 for sale under these specifications except where such pipe fails to comply with the weight requirements alone, in which case it may be sold under the weight specifications with which it does comply.

SUPPLEMENTARY REQUIREMENTS FOR SEAMLESS PIPE FOR USE IN CENTRAL STATIONS AT PRESSURES OF 400 LB. PER SQ. IN., OR OVER, AND TEMPERATURES OF 750 F. TO 1000 F., OR OTHER APPLICATIONS WHERE A SUPERIOR GRADE OF PIPE IS REQUIRED

S1. These requirements shall not be considered unless specified in the order, in which event the supplementary tests shall be made at the mill, unless otherwise agreed upon, at the purchaser's expense and witnessed by his inspector before shipment of the material.

Check Analysis

S2. Check analysis may be made on any length of pipe. Individual lengths failing to meet the chemical requirements specified in Section 6 shall be rejected.

Transverse Tension Tests

S3. Transverse tension tests may be made on specimens from both ends of each length of pipe. If the specimen from either end of any length fails to meet the physical properties specified in Section 9, that length shall be rejected.

Flattening Tests

S4. The flattening tests specified in Section 11 may be made on specimens from both ends of each length of pipe. Crop ends may be used. If the specimen from either end of any length fails to meet the specified requirements, that length shall be rejected.

Finish and Metal Structure

S5. The finished pipe shall be free from all immoderate slivers and depressions. The steel shall have a homogeneous structure as shown by the etching test in Paragraph S6.

Etching Tests

S6. Etching tests[1] may be made on sections from any pipe, and shall show sound and reasonably uniform material, free from injurious laminations, cracks and similar objectionable defects. Sections used for the flattening tests described in Section 11 may be used for the etching test. If the specimen from any length shows objectionable defects, that length may be rejected.

Rejection

S7. At the purchaser's discretion, if 10 per cent of the pipe in any one lot fail to conform to the requirements of Paragraphs S2 to S6, the entire lot

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[1] Pending development of etching methods applicable to the product covered by these specifications, it is recommended the Tentative Recommended Practice for a Macro-Etch Test for Steel described in the *Metals Handbook*, Am. Soc. for Metals, 1936 edition, p. 573, be followed.

may be rejected. Rejection of a lot shall be based on tests from not less than two pipes. A lot shall consist of all the pipe bought by a purchaser of the same size and wall thickness from any one heat.

EXPLANATORY NOTE

NOTE.—Pending the incorporation of high-temperature data in an appendix, which it is the purpose in due time to add, high-temperature properties, when it is desired that the same be indicated or specified, shall be a matter of agreement between the manufacturer and the purchaser. No high-temperature tests shall be required on the materials selected unless a matter of agreement between the manufacturer and the purchaser. When required, the short-time tests at elevated temperature shall be in accordance with the Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (A.S.T.M. Designation: E 21 – 37 T)[1] and the creep tests shall be in accordance with the Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (A.S.T.M. Designation: E 22 – 35 T)[2] of the American Society for Testing Materials.

APPENDIX

The following sizes and wall thicknesses of carbon-molybdenum pipe as provided for in these specifications were agreed on as standard for stock purposes by the pipe manufacturers and the Prime Movers Committee of the Edison Electric Institute. These data are included here for information only.

Nominal Pipe Size, in.	Service up to 1400 lb. per sq. in. at 950 F.		Service up to 900 lb. per sq. in. at 900 F.	
	Schedule No.	Thickness, in.	Schedule No.	Thickness, in.
$\frac{3}{4}$	160	0.218	160	0.218
$1\frac{1}{4}$	160	0.250	160	0.250
$2\frac{1}{2}$	160	0.375	160	0.375
4	160	0.531	120	0.437
6	160	0.718	120	0.562
8	160	0.906	100	0.593
10	160	1.125	100	0.718
12	160	1.312	100	0.843
14	160	1.406	100	0.937
16	160	1.562	100	1.031

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[1] See p. 1290.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, 1291 (1935); also 1937 Book of A.S.T.M. Tentative Standards, p. 1544.

TENTATIVE SPECIFICATIONS
FOR
ZINC-COATED (GALVANIZED) IRON OR STEEL BARBED WIRE[1]

A.S.T.M. Designation: A 121 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover three classes of zinc-coated iron or steel barbed wire (galvanized before fabrication), namely, Classes 1, 2, and 3 (as described in Section 6), as designated by the weight of coating in ounces of zinc per square foot of bare wire surface.

2. These specifications cover the usual sizes and standard constructions of barbed wire as follows:

Size, Steel Wire Gage	Nominal Diameter of Zinc-Coated Wire in Strand, in.	Commercial Designation	Number of Barbs	Barb Spacing, in.
No. 12	0.105	Hog	4	4
No. 12	0.105	Cattle	4	6
No. 12½	0.099	Hog	4	4
No. 12½	0.099	Cattle	4	6
No. 12½	0.099	Hog	2	3
No. 12½	0.099	Cattle	2	5
No. 14	0.080	Hog	2	3
No. 14	0.080	Cattle	2	5

Zinc Coating

3. The zinc used for the coating shall be any grade of zinc conforming to the requirements of the Standard Specifications for Slab Zinc (Spelter) (A.S.T.M. Designation: B 6) of the American Society for Testing Materials.[2]

Base Metal

4. The base metal of the barbed wire shall be of a good commercial quality of steel or iron wire. If copper steel wire is specified, the copper content shall conform to the following requirements:

Copper, when copper steel is specified, min., per cent	Ladle analysis	0.20
	Check analysis	0.18

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-5 on Corrosion of Iron and Steel.

These specifications are in effect a tentative revision of and are intended to supersede when adopted as standard, the present Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (A.S.T.M. Designation: A 121 – 30), 1936 Book of A.S.T.M. Standards, Part I, p. 362.

[2] 1937 Supplement to Book of A.S.T.M. Standards, p. 43.

Weight and Uniformity of Coating

5. The weight and uniformity of the coating for the various gages of wire composing the strands of the barbed wire shall be specified by the purchaser and shall be in accordance with the weights and Preece test dips for the particular class of coating specified as designated in Table I and Table II.

Tests of Coating

6. (*a*) *Weight of Coating.*—The weight of the zinc coating (ounces per square foot) shall be determined in accordance with the Standard Methods of Determining Weight of Coating on Zinc-Coated (Galvanized) Iron or Steel Articles (A.S.T.M. Designation: A 90) of the American Society for Testing Materials.[1]

TABLE I.—WEIGHT OF COATING IN OUNCES PER SQUARE FOOT OF BARE WIRE SURFACE.

Size, Steel Wire Gage	Nominal Diameter of Zinc-Coated Wire in Strand, in.	Minimum Weight of Coating		
		Class 1	Class 2	Class
No. 12....................	0.105	0.30	0.50	0.80
No. 12½..................	0.099	0.30	0.50	0.80
No. 14....................	0.080	0.25	0.45	0.65

TABLE II.—UNIFORMITY OF COATING (PREECE TEST).

Size, Steel Wire Gage	Minimum Number of Preece Test Dips		
	Class 1	Class 2	Class 3
No. 12..................................	1½	2	4
No. 12½................................	1½	2	3
No. 14..................................	1	1½	2

(*b*) *Uniformity of Coating.*—The uniformity of the zinc coating shall be determined by the Preece test in accordance with the Tentative Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Wire (A.S.T.M. Designation: A 191 - 36 T) of the American Society for Testing Materials.[2]

Sampling and Number of Tests

7. For purpose of tests, one spool shall be selected at random from every 50 spools or fraction thereof in a shipment. Sufficient length of barbed wire shall be cut from the end of the spool to provide material for the tests prescribed in Sections 5 and 6.

Retests and Rejection

8. If 20 per cent of the samples tested for the weight of zinc coating or the uniformity of zinc coating fail to conform to the requirements of these specifications, then an additional test of four other spools, selected

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 397.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 645 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 237.

from each lot of 50 spools or fraction thereof, shall be made and, if 20 per cent of the total samples fail to conform to the requirements, then the entire lot represented by the samples may be rejected.

Size and Permissible Variations

9. (*a*) The size of the zinc-coated wire shall be expressed in terms of the gage numbers of the steel wire gage given in Table I.

(*b*) The permissible variations from the nominal diameter of barbed wire shall be ±0.005 in.

Packing and Marking

10. Each spool of barbed wire shall be compactly wound and fastened. Each spool shall be legibly marked with the manufacturer's name, style of barbed wire, class of coating, and A.S.T.M. serial designation A 121.

Inspection

11. The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests and inspection shall be made at the place of manufacture prior to shipment, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Billing

12. All spools of barbed wire accepted by the purchaser shall be billed to him on the basis of the original weight or length on the spools before sampling.

TENTATIVE SPECIFICATIONS
FOR
ZINC-COATED (GALVANIZED) IRON OR STEEL FARM-FIELD AND RAILROAD RIGHT-OF-WAY WIRE FENCING[1]

A.S.T.M. Designation: A 116 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover three classes of zinc-coated iron or steel wire fencing (galvanized before fabrication), for farm-field fence and railroad right-of-way fence, namely, Classes 1, 2, and 3 (as described in Section 6), as designated by the weight of coating in ounces of zinc per square foot of bare wire surface.

Style and Size

2. It is recommended that styles and sizes of fence fabrics covered by these specifications be in accordance with those recommended by the U. S. Department of Commerce in its Simplified Practice Recommendation No. 9 or its subsequent recommendations.

Zinc Coating

3. The zinc used for the coating shall be any grade of zinc conforming to the requirements of the Standard Specifications for Slab Zinc (Spelter) (A.S.T.M. Designation: B 6) of the American Society for Testing Materials.[2]

Base Metal

4. The base metal of the fabric shall be of a good commercial quality of steel or iron wire. If copper steel wire is specified, the copper content shall conform to the following requirements:

Copper, when copper steel is specified, min., per cent	Ladle analysis	0.20
	Check analysis	0.18

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-5 on Corrosion of Iron and Steel.

These specifications are in effect a tentative revision of, and are intended to supersede when adopted as standard, the present Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (A.S.T.M. Designation: A 116 – 30), 1936 Book of A.S.T.M. Standards, Part I, p. 368.

[2] 1937 Supplement to Book of A.S.T.M. Standards, p. 43.

Weight and Uniformity of Coating

5. The weight and uniformity of the coating for the various gages of wire composing the fencing shall be specified by the purchaser and shall be in accordance with the weights and Preece test dips for the particular class of coatings specified as designated in Tables I and II.

TABLE I.—WEIGHT OF COATING IN OUNCES PER SQUARE FOOT OF BARE WIRE SURFACE.

Size, Steel Wire Gage	Nominal Diameter of Zinc-Coated Wire, in.	Minimum Weight of Coating Class 1	Class 2	Class 3
No. 7	0.177	0.40	0.60	0.80
No. 9	0.148	0.40	0.60	0.80
No. 10	0.135	0.30	0.50	0.80
No. 11	0.120	0.30	0.50	0.80
No. 12	0.105	0.30	0.50	0.80
No. 12½	0.099	0.30	0.50	0.80

TABLE II.—UNIFORMITY OF COATING (PREECE TEST).

Size, Steel Wire Gage	Minimum Number of Preece Test Dips Class 1	Class 2	Class 3
Nos. 7 and 9	2½	3	4
Nos. 10 to 12, incl	1½	2	4
No. 12½	1½	2	3

Tests of Coating

6. (*a*) *Weight of Coating.*—The weight of the zinc coating (ounces per square foot) shall be determined in accordance with the Standard Methods of Determining Weight of Coating on Zinc-Coated (Galvanized) Irno or Steel Articles (A.S.T.M. Designation: A 90) of the American Society for Testing Materials.[1]

(*b*) *Uniformity of Coating.*—The uniformity of the zinc coating shall be determined by the Preece test in accordance with the Tentative Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Wire (A.S.T.M. Designation: A 191 - 36 T) of the American Society for Testing Materials.[2]

Sampling and Number of Tests

7. For purpose of tests, one roll from each lot of 50 rolls of fencing, or fraction thereof. in a shipment, shall be selected for a sample. Sufficient length of the fencing shall be cut from the end of the roll to include three stay wires. The test specimens for the line wire shall be cut between these three stays. The test specimens for the stays shall be cut from two of these three stays. All wire knots, wraps or welds shall be excluded from the test. The resultant specimens of line and stay wire from each sample roll shall be put into separate groups. Three specimens from each of these groups shall be subjected to the test for the weight of coating in accordance with Sections 5 and 6, and three specimens from each of these groups shall be subjected to the test for uniformity of coating, in accordance with Sections 5 and 6.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 397.

[2] *Proceedings*, Am. Soc. Testing Mats. Vol. 36, Part I, p. 645 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 237.

Retests and Rejection

8. If 20 per cent of the specimens tested for the weight of zinc coating, or the uniformity of zinc coating, fail to meet the requirements of these specifications, four additional rolls shall be selected, samples taken as described above, and a retest for the non-conforming properties shall be made; and if 20 per cent of the total number of specimens fail, the entire lot represented by the samples may be rejected.

Size and Permissible Variations

9. (*a*) The size of the zinc-coated wire shall be expressed in terms of the gage numbers of the steel wire gage given in Table I.

(*b*) The permissible variations from the nominal diameter shall be as follows:

SIZE, STEEL WIRE GAGE	PERMISSIBLE VARIATIONS FROM NOMINAL DIAMETER
Nos. 7 to 12, incl	±0.004 in.
No. 12½	±0.003 in.

Packing and Marking

10. Each roll of wire fencing shall be tightly rolled and firmly tied. Each roll shall carry a tag showing the style of fence, the length of fencing in the roll, class of coating, A.S.T.M. serial designation A 116, and the manufacturer's name or mark.

Inspection

11. The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests and inspection shall be made at the place of manufacture prior to shipment, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

Billing

12. All rolls of fencing accepted by the purchaser shall be billed on the basis of the original weight or length of the rolls before sampling.

TENTATIVE SPECIFICATIONS
FOR
SINGLE AND DOUBLE REFINED WROUGHT-IRON BARS[1]

A.S.T.M. Designation: A 189 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1936; Revised, 1937.

Scope

1. These specifications cover refined wrought-iron bars, as follows:

Grade A.—Double refined, round, hexagonal and rectangular bars.

Grade B.—Single refined, round, hexagonal and rectangular bars.

Manufacture

2. (*a*) The bars shall be made from pig puddled or processed wrought iron[2] and shall be free from any admixture of iron scrap or steel. In the final piling all bars shall be of the full length of the pile.

Grade A.—The original muck bars shall be piled, rolled, repiled and rerolled.

Grade B.—The original muck bars shall be piled and rolled.

(*b*) *Iron Scrap.*—Unless otherwise specified by the purchaser, the term "iron scrap," applies only to foreign or bought scrap and does not include local mill products which are free from foreign or bought scrap. Any local mill products used shall be the equal of the material specified.

Chemical Composition

3. The iron shall conform to the following requirements as to chemical composition:

Manganese, max., per cent.. 0.06

Check Analyses

4. (*a*) An analysis may be made by the purchaser from a broken tension test specimen representing each lot as specified in Section 9 (*a*). The chemical composition thus determined shall conform to the requirement specified in Section 3.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-2 on Wrought Iron.

[2] Wrought iron is defined as follows in the Standard Definitions of Terms Relating to Wrought Iron Specifications (A.S.T.M. Designation: A 81) of the American Society for Testing Materials:

Wrought Iron.—A ferrous material, aggregated from a solidifying mass of pasty particles of highly refined metallic iron with which, without subsequent fusion, is incorporated a minutely and uniformly distributed quantity of slag.

(*b*) Drillings for analysis shall be so taken as to represent the entire cross-section of the specimen.

Tension Tests

5. (*a*) The iron shall conform to the following requirements as to tensile properties:

	Round, Square and Hexagonal Bars under $1\frac{5}{8}$ in. in Diameter or Thickness	Round, Square and Hexagonal Bars over $1\frac{5}{8}$ in. and up to but not including $2\frac{1}{2}$ in. in Diameter or Thickness	Round, Square and Hexagonal Bars $2\frac{1}{2}$ in. and over in Diameter or Thickness, and all Flat Bars
GRADE A—DOUBLE REFINED			
Tensile strength, lb. per sq. in[b]	48 000 to 54 000	47 000 to 54 000	46 000 to 54 000
Yield point, min., lb. per sq. in.	0.6 tens. str.	0.55 tens. str.	0.50 tens. str.
Elongation in 8 in., min., per cent	28[a]	25	22
Reduction of area, min., per cent	45	40	35
GRADE B—SINGLE REFINED			
Tensile strength, min., lb. per sq. in[b]	48 000	47 000	46 000
Yield point, min., lb. per sq. in.	0.6 tens. str.	0.55 tens. str.	0.50 tens. str.
Elongation in 8 in., min., per cent	25[a]	22	20
Reduction of area, min., per cent	40	35	30

[a] Bars not exceeding $\frac{7}{8}$ in. in diameter or thickness will be accepted if the elongation in 8 in. is not less than 25 per cent for Grade A and 22 per cent for Grade B material.

[b] A deduction of 1000 lb. per sq. in. shall be allowed from the minimum tensile strength specified for specimens reduced by machining.

(*b*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine. The speed of the crosshead of the machine shall not exceed $\frac{3}{4}$ in. per min. After passing the yield point, the speed of the crosshead shall not exceed 3 in. per min.

Bend Tests

6. (*a*) *Cold Bend Tests.*—The test specimen shall stand being bent cold through 180 deg. around a pin the diameter of which is equal to the diameter or thickness of the specimen, without fracture on the outside of the bent portion.

(*b*) *Hot Bend Tests.*—The test specimen when heated to a temperature between 1700 and 1800 F. (925 and 980 C.), shall stand being bent through 180 deg. flat on itself, without fracture on the outside of the bent portion.

(*c*) *Nick Bend Tests.*—The test specimen, when nicked 25 per cent around with a tool having a 60-deg. cutting edge to a depth of not less than 8 nor more than 16 per cent of the diameter or thickness of the specimen and broken slowly, shall show a wholly fibrous fracture.

(*d*) Bend tests may be made by pressure or by blows.

Step Test

7. In the case of Grade A bars only, the test specimen when reduced to 50 per cent of its original diameter by smooth-turned steps, each 2 in. in length and $\frac{1}{8}$ in. less in diameter than the preceding, shall indicate freedom from injurious seams, laminations and other internal defects.

Etch Test

8. The cross section of the test specimen shall be ground or polished and etched[1] for a sufficient period to develop the structure. This test shall show the material to be uniform and free from steel.

Test Specimens

9. (*a*) *Tension Test Specimens.*—Tension test specimens shall be the full section of material as rolled, if possible, otherwise, the specimens shall be taken from the material as rolled. For bars 2½ in. or less in diameter or thickness, the axis of the tension specimen shall coincide with the axis of the bar. For bars over 2½ in. in diameter or thickness, the axis of the tension specimen shall be located at any point one-half the distance from the center to the surface and shall be parallel to the axis of the bar; and the specimen may be turned to a convenient diameter for a length of at least 9 in. with enlarged ends.

(*b*) *Bend Test Specimens.*—Round, square and hexagonal bars not over 1½ in. in diameter or thickness, and flat bars not over 1 in. in thickness or 2 in. in width shall be bent in full section as rolled. For larger round, square and hexagonal bars the bend specimen may be reduced to 1½ in. in diameter. For flat bars the bend specimens may be reduced to a width not less than the thickness of the bar. In any case, however, it is permissible to reduce the bend test specimen for flat bars to at least a one inch square. The edges of the machined specimens shall be rounded to a radius of $\frac{1}{16}$ in.

Number of Tests

10. (*a*) Bars of each size shall be sorted into lots of 100 each. Two bars shall be selected at random from each lot or fraction thereof, and tested as specified in Sections 4, 5, 6 and 7; but only one of these bars shall be tested as specified in Section 8.

(*b*) Where small lots of different sizes are to be tested the bars shall be grouped into lots of 5000 lb., or fraction thereof, without regard to size, and one bar representing each lot of 5000 lb., or fraction thereof, shall be tested as specified in Sections 4, 5, 6, 7 and 8.

(*c*) If any of the specimens originally selected to represent a lot of material contain surface defects not visible before testing but visible after testing, or if a tension test specimen fails to meet the physical requirements of Section 5 and breaks outside the middle third of the gage length, as indicated by scribe scratches marked on the specimen before testing, the individual specimen shall be rejected and one retest from a different bar will be allowed.

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[1] A solution of 10 per cent hydrochloric acid (sp. gr. 1.19), 30 per cent sulfuric acid (sp. gr. 1.89), and 60 per cent water; or 25 per cent nitric acid (sp. gr. 1.42) and 75 per cent water, is recommended for the etch test.

Permissible Variations in Size

11. (*a*) Round, square and hexagonal bars shall not vary more than the limits given in the following table:

Nominal Size of Round, Square and Hexagonal Bars	Permissible Variations in Size, in.	
	Under	Over
Up to ½ in., incl	0.007	0.007
Over ½ in. up to 1 in., incl	0.10	0.10
Over 1 in. up to 2 in., incl	$\frac{1}{64}$	$\frac{1}{32}$
Over 2 in. up to 3 in., incl	$\frac{1}{32}$	$\frac{3}{64}$
Over 3 in. up to 5 in., incl	$\frac{1}{32}$	$\frac{3}{32}$
Over 5 in. up to 8 in., incl	$\frac{1}{16}$	$\frac{1}{8}$

(*b*) The width or thickness of flat bars shall not vary more than 2 per cent from that specified.

Finish

12. The bars shall be smoothly rolled and free from slivers, depressions, seams, crop ends and evidences of being burnt.

Marking

13. The bars shall be stamped or marked as designated by the purchaser.

Inspection

14. (*a*) The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified.

(*b*) The purchaser may make the tests to govern the acceptance or rejection of material in his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

Rejection

15. (*a*) If either of the test bars selected to represent a lot does not conform to the requirements specified, the lot will be rejected.

(*b*) Bars which develop defects in forging or machining will be rejected, and the manufacturer shall be notified.

Rehearing

16. Samples tested in accordance with Section 14 (*b*), which represent rejected material shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS
FOR
WROUGHT-IRON PLATES[1]

A.S.T.M. Designation: A 42 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ADOPTED, 1913; REVISED, 1918, 1930; REISSUED AS TENTATIVE, 1934;
REVISED, 1935, 1936, 1937.

Scope

1. These specifications cover standard and special forming wrought-iron[2] plates for general fabrication purposes, including bending and flanging, in thicknesses $\frac{3}{16}$ in. and over.

Manufacture

2. Plates shall be rolled from blooms, piles or slabs made from all pig puddled or processed wrought iron, free from any admixture of iron scrap (Note) or steel. The component parts of piles shall be of the full length of the pile.

NOTE.—The term iron scrap applies only to foreign or bought scrap and does not include local mill products which are free from foreign or bought scrap. Any local mill products used shall be the equal or of a higher grade than the material specified.

Chemical Composition

3. The iron shall conform to the following requirement as to chemical composition:

Manganese, max., per cent.................................... 0.06

Tension Tests

4. (*a*) *Standard Plate.*—Standard plate shall conform to the following minimum requirements as to tensile properties. These

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-2 on Wrought Iron.

These tentative specifications are in effect a tentative revision of, and replace the former Standard Specifications for Wrought-Iron Plates (A.S.T.M. Designation: A 42 - 30), which standard was accordingly discontinued in 1936.

[2] Wrought iron is defined as follows in the Standard Definitions of Terms Relating to Wrought Iron Specifications (A.S.T.M. Designation: A 81) of the American Society for Testing Materials:

Wrought Iron.—A ferrous material, aggregated from a solidifying mass of pasty particles of highly refined metallic iron with which, without subsequent fusion, is incorporated a minutely and uniformly distributed quantity of slag.

requirements apply only to longitudinal test specimens; no transverse tests shall be made:

Tensile strength, lb. per sq. in.	48 000
Yield point, lb. per sq. in.	27 000
Elongation in 8 in., per cent	14

(*b*) *Special Forming Plate.*—With special forming plate, the longitudinal strength and ductility are directly related to the transverse properties, and are scaled downward in ratio to the increase in transverse properties, according to the following formulas:

Tensile strength, lb. per sq. in. = 51,000 − (1500 × transverse ductility of plate (expressed as percentage elongation in 8 in.)

Longitudinal elongation in 8 in., per cent = 16 − transverse ductility (expressed as percentage elongation in 8 in.)

Yield point, lb. per sq. in. 27,000

(*c*) The purchaser shall specify in the order the minimum transverse ductility desired in conformity with the fabrication requirements. When no transverse ductility is specified in the order, standard plate will be supplied.

(*d*) Representative requirements for special forming plate, in accordance with the formulas, are as follows:

TRANSVERSE DUCTILITY (ELONGATION IN 8 IN.), PER CENT		MINIMUM LONGITUDINAL PROPERTIES		
		TENSILE STRENGTH,[a] MIN., LB. PER SQ. IN.		ELONGATION[b] IN 8 IN., MIN., PER CENT
3	51,000 − (1500 × 3) =	46 500	(16 − 3) =	13
4		45 000		12
5		43 500		11
6		42 000		10
7		40 500		9
8		39 000		8

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[a] See Section 5 (*a*).
[b] See Section 5 (*b*).

(*e*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine. The speed of the crosshead of the machine shall not exceed $\frac{3}{4}$ in. per minute.

Modifications in Elongation

5. (*a*) For plates over $\frac{3}{4}$ in. in thickness, a reduction of 1000 lb. per sq. in. from the indicated tensile strength will be allowed for each increase of $\frac{1}{8}$ in. in thickness over $\frac{3}{4}$ in., with a proportional reduction for fractional parts of $\frac{1}{8}$ in.; but in no case shall the tensile strength be less than 39,000 lb. per sq. in.

(*b*) For plates under $\frac{7}{16}$ in. in thickness, a deduction of 1 per cent from the percentages of elongation specified in Section 4 shall be made for each decrease of $\frac{1}{16}$ in. in thickness below $\frac{7}{16}$ in.; but in no case shall the elongation be less than 8 per cent.

Bend Tests

6. (*a*) *Cold-bend Tests.*—The test specimen shall stand being bent cold through 90 deg., without fracture on the outside of the bent portion, around a pin the diameter of which is:

$$d = \frac{40 \times \text{thickness of plate}}{\text{specified longitudinal elongation in 8 in., per cent}}$$

(*b*) *Nick-bend Tests.*—The test specimen, when nicked on one side and slowly broken, shall show a wholly fibrous fracture.

Test Specimens

7. (*a*) Tension and bend test specimens shall be taken from the finished plate and except as specified in Paragraph (*b*) shall be of the full thickness of the plate as rolled.

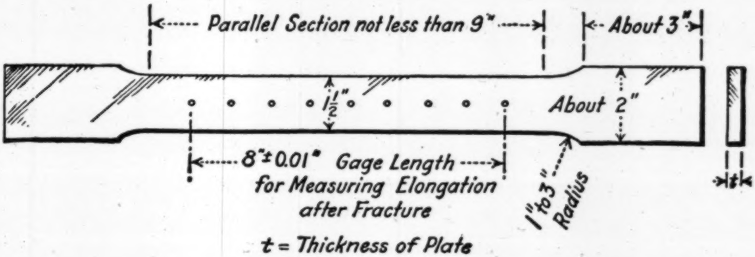
Parallel Section not less than 9"
About 3"
1½"
About 2"
8" ± 0.01" Gage Length for Measuring Elongation after Fracture
1" to 3" Radius
t
t = Thickness of Plate

FIG. 1.—Standard 8-in. Gage Length Test Specimen.

(*b*) Test specimens may be machined to the form and dimensions shown in Fig. 1, or with both edges parallel. When plates are 1½ in. or over in thickness, the specimens may be machined to a thickness of not less than ¾ in. or, if desired, to a diameter of not less than ¾ in. for a distance of not less than 9 in.

(*c*) The machined edges of rectangular bend test specimens may be rounded to a radius of not over $\frac{1}{16}$ in.

Number of Tests

8. (*a*) One tension, one cold-bend and one nick-bend test shall be made for each ⅛-in. variation in plate thickness and not less than one test for every ten plates as rolled. Where modification of the usual transverse properties is specified, and the tensile properties are determined by the formulas in Section 4 (*a*), two tension test specimens shall be taken at right angles to each other, one parallel to the longitudinal axis and the other parallel to the transverse axis.

TABLE I.—PERMISSIBLE VARIATIONS OF RECTANGULAR PLATES ORDERED TO WEIGHT.

Ordered Weight, lb. per sq. ft.	Permissible Variations in Average Weights per Square Foot of Plates for Widths Given, Expressed in Percentages of Ordered Weights																		Ordered Weight, lb. per sq. ft.
	Under 48 in.		48 to 60 in., excl.		60 to 72 in., excl.		72 to 84 in., excl.		84 to 96 in., excl.		96 to 108 in., excl.		108 to 120 in., excl.		120 to 132 in., excl.		132 in. or over		
	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	
Under 5	5	3	5.5	3	6	3	7	3	...	...	...	...	...	...	...	...	...	...	Under 5
5 to 7.5 excl.	4.5	3	5	3	5.5	3	6	3	...	...	...	...	...	...	...	...	...	...	5 to 7.5 excl.
7.5 " 10 "	4	3	4.5	3	5	3	5.5	3	6	3	7	3	8	3	...	...	...	...	7.5 " 10 "
10 " 12.5 "	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	7	3	8	3	9	3	10 " 12.5
12.5 " 15 "	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	7	3	8	3	12.5 " 15
15 " 17.5 "	2.5	2.5	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	7	3	15 " 17.5
17.5 " 20 "	2.5	2	2.5	2.5	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	6	3	17.5 " 20 "
20 " 25 "	2	2	2.5	2	2.5	2.5	3	2.5	3.5	2.5	4	3	4.5	3	5	3	5.5	3	20 " 25 "
25 " 30 "	2	2	2	2	2.5	2	2.5	2.5	3	2.5	3.5	3	4	3	4.5	3	5	3	25 " 30
30 40 "	2	2	2	2	2	2	2.5	2	2.5	2.5	3	2.5	3.5	3	4	3	4.5	3	30 " 40
40 or over	2	2	2	2	2	2	2	2	2.5	2	2.5	2.5	3	2.5	3.5	3	4	3	40 or over

NOTE.—The weight per square foot of individual plates shall not vary from the ordered weight by more than one and one-third the amount given in this table.

TABLE II.—PERMISSIBLE OVERWEIGHTS OF RECTANGULAR PLATES ORDERED TO THICKNESS.

Ordered Thickness, in.	Permissible Excess in Average Weights per Square Foot of Plates for Widths Given, Expressed in Percentages of Nominal Weights									Ordered Thickness, in.
	Under 48 in.	48 to 60 in., excl.	60 to 72 in., excl.	72 to 84 in., excl.	84 to 96 in., excl.	96 to 108 in., excl.	108 to 120 in., excl.	120 to 132 in., excl.	132 in. or over	
Under $\frac{1}{8}$	9	10	12	14	....	....	..	..	..	Under $\frac{1}{8}$
$\frac{1}{8}$ to $\frac{3}{16}$ excl.	8	9	10	12	....	....	..	..	..	$\frac{1}{8}$ to $\frac{3}{16}$ excl.
$\frac{3}{16}$ " $\frac{1}{4}$ "	7	8	9	10	12	....	..	..	..	$\frac{3}{16}$ " $\frac{1}{4}$ "
$\frac{1}{4}$ " $\frac{5}{16}$ "	6	7	8	9	10	12	14	16	19	$\frac{1}{4}$ " $\frac{5}{16}$
$\frac{5}{16}$ " $\frac{3}{8}$ "	5	6	7	8	9	10	12	14	17	$\frac{5}{16}$ " $\frac{3}{8}$ "
$\frac{3}{8}$ " $\frac{7}{16}$ "	4.5	5	6	7	8	9	10	12	15	$\frac{3}{8}$ " $\frac{7}{16}$ "
$\frac{7}{16}$ " $\frac{1}{2}$ "	4	4.5	5	6	7	8	9	10	13	$\frac{7}{16}$ " $\frac{1}{2}$ "
$\frac{1}{2}$ " $\frac{5}{8}$ "	3.5	4	4.5	5	6	7	8	9	11	$\frac{1}{2}$ " $\frac{5}{8}$ "
$\frac{5}{8}$ " $\frac{3}{4}$ "	3	3.5	4	4.5	5	6	7	8	9	$\frac{5}{8}$ " $\frac{3}{4}$ "
$\frac{3}{4}$ " 1 "	2.5	3	3.5	4	4.5	5	6	7	8	$\frac{3}{4}$ " 1 "
1 or over	2.5	2.5	3	3.5	4	4.5	5	6	7	1 or over

NOTE.—The weight of individual plates ordered to thickness shall not exceed the nominal weight by more than one and one-third the amount given in this table.

(*b*) If any of the specimens originally selected to represent a lot of material contains surface defects not visible before testing but visible after testing, or if a tension test specimen fails to meet the physical requirements of Section 4 and breaks outside the middle third of the gage length, as indicated by scribe scratches marked on the specimen before testing, the individual specimen shall be rejected and one retest from a different plate will be allowed. If the retest also fails, the plates represented by such test will be rejected.

Other Tests

9. Where desired, the purchaser may, at his own expense, subject the material to chemical, microscopic, or other tests to determine its quality in conformity with the intent of these specifications.

Variations in Weight and Thickness

10. The weight or thickness of plates shall be covered by the following permissible variations. One cubic inch of wrought iron is assumed to weigh 0.2776 lb.

(*a*) *Plates, When Ordered to Weight per Square Foot.*—The weight of each lot[1] of plates in each shipment shall not vary from the weight ordered more than the amounts given in Table I.

(*b*) *Plates, When Ordered to Thickness.*—The thickness of each plate shall not vary more than 0.01 in. under that ordered.

The overweight in each lot[1] of plates in each shipment shall not exceed the amounts given in Table II.

Finish

11. The plates shall be straight, smooth and free from cinder spots and holes, injurious flaws, buckles, blisters, seams and laminations.

Marking

12. The plates shall be stamped or otherwise marked as designated by the purchaser.

Inspection

13. (*a*) The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the plates ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to

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[1] The term "lot" as applied to Table I means all the plates of each group width and group weight. As applied to Table II, it means all the plates of each group width and group thickness.

satisfy him that the plates are being furnished in accordance with these specifications. All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

(*b*) The purchaser may make the tests to govern the acceptance or rejection of plates at his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

Rejection

14. (*a*) Unless otherwise specified, any rejection based on tests made in accordance with Section 13 (*b*) shall be reported within five working days from the receipt of samples.

(*b*) Material which shows injurious defects subsequent to its acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified.

Rehearing

15. Samples tested in accordance with Section 13 (*b*), which represent rejected plates, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS
FOR
WROUGHT-IRON RIVETS AND RIVET ROUNDS[1]

A.S.T.M. Designation: A 152 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1933; REVISED, 1935, 1936, 1937.

A. Requirements for Rolled Bars

MANUFACTURE

Process

1. The rivet bars shall be made from all pig puddled or processed wrought iron,[2] and shall be free from any admixture of iron scrap or steel.

NOTE.—The term "iron scrap" applies only to foreign or bought scrap and does not include local mill products which are free from foreign or bought scrap. Any local mill products used shall be the equal or of a higher grade than the material specified.

CHEMICAL PROPERTIES AND TESTS

Chemical Composition

2. The wrought iron shall conform to the following requirement as to chemical composition:

Manganese, max., per cent.................................. 0.06

Check Analyses

3. (*a*) Analyses may be made by the purchaser from a broken test specimen representing each lot, as specified in Section 7 (*a*). At the discretion of the purchaser, the chemical analysis may be made from a rivet. Each rivet so tested shall not represent more than 2000 lb. of any one size.

(*b*) Drillings for analysis shall be so taken as to represent the entire cross-section of the specimen.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-2 on Wrought Iron.

[2] Wrought iron is defined as follows in the Standard Definitions of Terms Relating to Wrought Iron Specifications (A.S.T.M. Designation: A 81) of the American Society for Testing Materials:

Wrought Iron.—A ferrous material, aggregated from a solidifying mass of pasty particles of highly refined metallic iron, with which, without subsequent fusion, is incorporated a minutely and uniformly distributed quantity of slag.

PHYSICAL PROPERTIES AND TESTS

Tension Tests

4. (*a*) The wrought iron shall conform to the following minimum requirements as to tensile properties:

Tensile strength, lb. per sq. in.		47 000
Yield point, lb. per sq. in		0.6 tens. str.
Elongation in 8 in., per cent	rounds $\frac{1}{4}$ to $\frac{7}{16}$ in., incl., in diam	22
	rounds $\frac{7}{16}$ to $\frac{3}{4}$ in., incl., in diam	24
	rounds over $\frac{3}{4}$ to 2 in., incl., in diam	28

(*b*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine.

Bend Tests

5. (*a*) *Quenched, Cold-bend Tests.*—A test specimen shall be heated between 1600 and 1700 F. (870 and 925 C.), and quenched in cold water, after which it shall stand being bent cold through 90 deg. on a radius which is equal to the diameter of the specimen without fracture on the outside of the bent portion.

(*b*) *Nick-bend Tests.*—A test specimen, when nicked 25 per cent around the circumference with a tool having a 60-deg. cutting edge, to a depth of not less than 8 nor more than 16 per cent of the diameter or thickness of the specimen, and broken slowly, shall show a wholly fibrous fracture.

(*c*) *Hot-bend Tests.*—A test specimen, when heated to a temperature between 1700 and 1800 F. (925 and 980 C.), shall stand being bent through 180 deg. flat on itself without fracture on the outside of the bent portion.

(*d*) Bend tests may be made by pressure or blows.

Test Specimens

6. All test specimens shall be of the full section of the material as rolled.

Number of Tests

7. (*a*) All bars of a given size shall be piled separately, sorted in lots of 100 each. Two bars shall be selected at random from each lot or fraction thereof and tested as specified in Sections 4 to 6.

(*b*) If any of the specimens originally selected to reprenes a lot of material contains surface defects not visible before testing but visible after testing, or if a tension test specimen fails to meet the physical test requirements of Section 4 and breaks outside the middle third of the gage length, as indicated by scribe scratches marked on the specimen before testing, the individual specimen shall be rejected and one retest from a different bar will be allowed.

Micrographic Examination

8. In case of doubt as to the presence of steel, the purchaser may, at his own expense, make micrographic examination to assist in determining whether the material conforms to the requirements of Section 1.

Permissible Variations

9. The diameter of rivet bars shall not vary from that specified by more than the amount given in the following table:

DIAMETER, IN.	PERMISSIBLE VARIATIONS IN DIAMETER, IN.	OUT OF ROUND, IN.
Up to ½, inclusive	±0.007	0.010
Over ½ to 1, inclusive	±0.010	0.013
Over 1 to 1¼, inclusive	±0.012	0.016
Over 1¼ to 1½, inclusive	±0.015	0.021
Over 1½ to 2, inclusive	±0.022	0.023

Finish

10. The bars shall be smoothly rolled and shall be free from slivers, depressions, crop ends and seams, and shall show no evidence of being burnt.

INSPECTION AND REJECTION

Inspection

11. (*a*) The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the bars are being furnished in accordance with these specifications. Tests and inspection at the place of manufacture shall be made prior to shipment.

(*b*) The purchaser may make the tests to govern the acceptance or rejection of material in his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

(*c*) All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection

12. (*a*) All bars of one size in any one lot will be rejected if the test specimens representing that size and lot do not conform to the requirements specified.

(*b*) Unless otherwise specified, any rejection based on tests made in accordance with Section 11 (*b*) shall be reported within five working days from the receipt of samples.

Rehearing

13. Samples tested in accordance with Section 11 (*b*), which represent rejected material, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

B. Requirements for Finished Rivets

Material

14. The material used in the manufacture of the rivets shall conform to the requirements for rolled bars as specified in Sections 1 to 13, inclusive.

PHYSICAL PROPERTIES AND TESTS

Flattening Test

15. The rivet head shall stand being flattened, while hot, to a diameter two times the diameter of the shank, as shown in Fig. 1, without cracking at the edges.

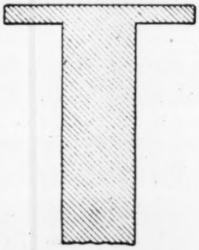

FIG. 1.—Flattening Test.

Etch Test

16. The rivet shall be split longitudinally through head and shank. After grinding or polishing and etching in acid,[1] the section shall show freedom of folds at base of head, and the fiber lines shall show symmetrical displacement. (See Fig. 2).

Number of Tests

17. Three flattening and three etch tests shall be made from each lot of 2000 lb. or less of any one diameter of rivets offered for inspection, each of which shall conform to the requirements specified in Sections 15 and 16.

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[1] A solution of 10 per cent hydrochloric acid (sp. gr. 1.19), 30 per cent sulfuric acid (sp. gr. 1.84), and 60 per cent water; or 25 per cent nitric acid (sp gr 1.42) and 75 per cent water, is recommended for the etch test.

WORKMANSHIP AND FINISH

Workmanship and Finish

18. The rivets shall be true to form and concentric, shall be made in a workmanlike manner, and shall be free from injurious defects. The heads shall not show cracks or splits on the edge but may contain a slight flash.

INSPECTION AND REJECTION

Inspection

19. The inspector representing the purchaser shall have free entry, at all times while the work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the rivets ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the rivets are being furnished in accordance with these specifications. All tests and inspection shall be made at the place of manufacture of the rivets prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

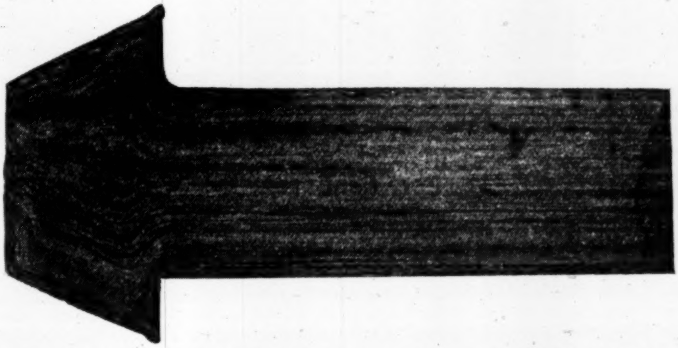

FIG. 2.—Showing Symmetrical Displacement of Fiber Lines After Etching Test.

Rejection

20. Rivets which show defects subsequent to their acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified.

TENTATIVE SPECIFICATIONS
FOR
ROLLED WROUGHT-IRON SHAPES AND BARS[1]

A.S.T.M. Designation: A 207 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.[2]

Scope

1. These specifications cover a general group of rolled wrought-iron structural shapes, sections and bars.

Manufacture

2. The shapes, sections and bars may be direct rolled from billets made from pig puddled or processed wrought iron[3] and shall be free from any admixture of iron scrap or steel.

NOTE.—The term "iron scrap" applies only to foreign or bought scrap and does not include local mill products which are free from foreign or bought scrap. Any local mill products used shall be the equal or of a higher grade than the material specified.

Chemical Composition

3. The iron shall conform to the following requirement as to chemical composition:

Manganese, max., per cent	0.06

Check Analysis

4. An analysis may be made by the purchaser from a broken tension test specimen representing each lot as specified in Section 8 (*a*). The chemical composition thus determined shall conform to the requirement specified in Section 3.

Tension Tests

5. (*a*) The iron shall conform to the following minimum requirements as to tensile properties:

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee A-2 on Wrought Iron.

[2] Accepted for publication as tentative by Committee E-10 on Standards, August 26, 1937.

[3] Wrought iron is defined as follows in the Standard Definitions of Terms Relating to Wrought Iron Specifications (A.S.T.M. Designation: A 81) of the American Society for Testing Materials:

Wrought Iron.—A ferrous material, aggregated from a solidifying mass of pasty particles of highly refined metallic iron with which, without subsequent fusion, is incorporated a minutely and uniformly distributed quantity of slag.

	ROUND, SQUARE AND HEXAGONAL BARS UNDER $1\frac{5}{8}$ IN. IN DIAMETER OR THICKNESS	ROUND, SQUARE AND HEXAGONAL BARS OVER $1\frac{5}{8}$ IN. AND UP TO BUT NOT INCLUDING $2\frac{1}{2}$ IN. IN DIAMETER OR THICKNESS	ROUND, SQUARE AND HEXAGONAL BARS $2\frac{1}{2}$ IN. AND OVER IN DIAMETER OR THICKNESS; ALL FLAT BARS AND SHAPES
Tensile strength, min., lb. per sq. in.	48 000	47 000	46 000[a, b]
Yield point, min., lb. per sq. in....	0.60 tens. str.	0.55 tens. str.	0.50 tens. str.
Elongation in 8 in., min., per cent..	25	22	20
Reduction of area, min., per cent..	40	35	30[c]

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[a] When the sectional area exceeds 12 sq. in., the allowable minimum tensile strength shall be 45,000 lb. per sq. in.

[b] For sections less than $\frac{3}{16}$ in. in thickness, no tension tests will be required.

[c] For flat bars, shapes and sections less than $\frac{3}{8}$ in. in thickness, no reduction of area test will be required.

(*b*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine. The speed of the crosshead of the machine shall not exceed $\frac{3}{4}$ in. per min. before passing the yield point.

Bend Tests

6. (*a*) *Cold-Bend Tests.*—The test specimen shall stand being bent cold through 180 deg. around a pin, the diameter of which is equal to the thickness of the specimen, without fracture on the outside of the bend.

For sections less than $\frac{3}{16}$ in. in thickness, no bend test will be required.

(*b*) *Hot-Bend Tests.*—The test specimen, when heated to a temperature between 1700 and 1800 F. (925 to 980 C.) shall stand being bent through 180 deg. flat on itself without fracture on the outside of the bent portion.

(*c*) *Nick-Bend Tests.*—The specimen, when nicked 25 per cent around for round specimens and along one side for rectangular specimens to a depth of not less than 8 nor more than 16 per cent of the diameter or thickness of the specimen and broken slowly, shall show a wholly fibrous fracture.

(*d*) Bend tests may be made by pressure or by blows.

Test Specimens

7. Where practicable, tension and bend test specimens shall be the full section of material as rolled. Bars over 2 in. in diameter or thickness may be machined to a diameter of 1 in. for a length of at least 9 in. with enlarged ends. Test specimens for all structural shapes shall be machined from the rolled shapes into specimens of rectangular section. For flat bars the bend test specimen may be reduced to a width not less than the thickness of the bar; but in any case it is permissible to reduce the specimen to at least a one inch square. The edges of a machined specimen shall be rounded to a radius of $\frac{1}{16}$ in.

Number of Tests

8. (*a*) One test each of those noted in Sections 5 and 6 shall be made on representative samples from each lot of 100 shapes or bars. Where small lots of different sizes are to be tested, the material shall be grouped into lots of 5000 lb. or fraction thereof, without regard to size, and one test specimen representing each lot of 5000 lb. or fraction thereof shall be tested as specified in Sections 5 and 6.

(*b*) If any of the specimens originally selected to represent a lot of material contains surface defects not visible before testing but visible after testing, or if a tension test specimen fails to meet the physical requirements of Section 5 and breaks outside the middle third of the gage length, as indicated by scribe scratches marked on the specimen before testing, the individual specimen shall be rejected and one retest from a different bar will be allowed. If the retest also fails, the product represented by such test will be rejected.

Permissible Variations in Size and Weight

9. (*a*) Round, square, and hexagonal bars shall not vary more than the limits given in the following table:

NOMINAL SIZE, ROUND, SQUARE, AND HEXAGONAL BARS	PERMISSIBLE VARIATIONS IN SIZE, IN.	
	UNDER	OVER
Up to $\frac{1}{2}$ in., inclusive	0.007	0.007
Over $\frac{1}{2}$ in. up to 1 in., inclusive	0.010	0.010
Over 1 in. up to 2 in., inclusive	$\frac{1}{64}$	$\frac{1}{32}$
Over 2 in. up to 3 in., inclusive	$\frac{1}{32}$	$\frac{3}{64}$
Over 3 in. up to 5 in., inclusive	$\frac{1}{32}$	$\frac{3}{32}$
Over 5 in. up to 8 in., inclusive	$\frac{1}{16}$	$\frac{1}{8}$

(*b*) The width or thickness of flat bars shall not vary more than 2 per cent from that specified.

(*c*) *Shapes.*—The weights of each lot[1] of angles, tees, zees, channels, or other shapes in each shipment shall not vary from the weight ordered more than the amounts given in the following table:

SHAPES	THICKNESS, IN.	PERMISSIBLE VARIATIONS FROM NOMINAL WEIGHTS
Any dimension over $1\frac{1}{2}$ in	Over $\frac{3}{16}$	±4 per cent
All dimensions $1\frac{1}{2}$ in. and less	Over $\frac{3}{16}$	±5 per cent
Any dimension over $1\frac{1}{2}$ in	$\frac{3}{16}$ and less	±6 per cent
All dimensions $1\frac{1}{2}$ in. and less	$\frac{3}{16}$ and less	±7 per cent

Finish

10. The finished material shall be free from injurious defects and shall have a workmanlike finish.

Inspection

11. (*a*) The inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the production of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the product is being furnished in accordance with these specifications. All tests and inspection (except check analyses) shall be made at the place of manufacture prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

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[1] The term "lot" here means all of the shapes of each group dimension and group thickness shown in the table.

(*b*) The purchaser may make the tests to govern the acceptance or rejection of material in his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

Rejection

12. Material which shows injurious defects during fabrication subsequent to its acceptance at the manufacturer's works will be rejected, and the manufacturer shall be notified.

Rehearing

13. Samples tested in accordance with Section 11 (*b*), which represent rejected material, shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

TENTATIVE SPECIFICATIONS
FOR
ALUMINUM-BASE ALLOYS IN INGOT FORM FOR SAND-CASTINGS[1]

A.S.T.M. Designation: B 58 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1926; ADOPTED IN AMENDED FORM, 1931; REISSUED AS TENTATIVE, 1933; REVISED, 1937.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

Scope

1. These specifications cover commercial aluminum-base alloys in ingot form for remelting for the manufacture of sand castings. The specific gravity of these alloys does not exceed 3.0. Twelve alloys are specified and are designated Alloys C, CC, F, G, GG, H, J, JJ, K, L, M and N (see Explanatory Note).

Process

2. The alloys may be made by any approved process.

Quality

3. The material covered by these specifications shall be of uniform quality and shall be free from dross, slag, and other harmful contamination.

Chemical Composition

4. The alloys shall conform to the chemical composition requirements specified in Table I.

Sampling for Chemical Analysis

5. (*a*) The sample for determining the chemical composition shall be taken by sawing, drilling, or milling the sample ingot in such a manner as to be representative of its average cross-section.

(*b*) The saw, drill, or cutter used for taking the sample shall be thoroughly cleaned. No lubricant shall be used in the operation, and the sawings or metal chips shall be carefully treated with a magnet to remove any particles of iron introduced in taking the sample.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee B-7 on Light Metals and Alloys, Cast and Wrought.

These specifications are in effect a revision of and replace the former Standard Specifications for Aluminum-Base Sand-Casting Alloys in Ingot Form (A.S.T.M. Designation: B 58 – 31) which standard was accordingly discontinued in 1933.

Methods of Chemical Analysis

6. The chemical analysis shall be made in accordance with the Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys (A.S.T.M. Designation: B 40 – 36 T) of the American Society for Testing Materials.[1]

Number of Tests

7. If the ingots are shipped in carload lots of the same alloy, not less than five ingots shall be taken at random from the carload for sampling. If the shipment is in less than carload lots, one sample ingot shall be taken for each 6000 lb. (2721.6 kg.) or fraction thereof. When it is deemed necessary, a sample may be taken from each melt of 500 lb. (226.8 kg.) or more of the alloy.

TABLE I.—CHEMICAL COMPOSITIONS OF ALUMINUM-BASE ALLOY INGOTS FOR SAND-CASTINGS.

NOTE 1.—Where single limits are shown in this table, these indicate the maximum amount permitted.

NOTE 2.—Analysis shall regularly be made only for the elements specifically mentioned in this table. If, however, the presence of other elements is indicated in the course of routine analysis, further analysis shall be made to determine that the total of these other elements is not present in excess of the limits specified in the last column of the table.

NOTE 3.—The following applies to all specified limits in this table: For purposes of acceptance and rejection, an observed value or a calculated value obtained from analysis should be rounded off to the nearest unit in the last right-hand place of figures used in expressing the specified limit.

Alloy	Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Magnesium, per cent	Manganese, per cent	Zinc, per cent	Titanium, per cent	Nickel, per cent	Total Other Impurities, max., per cent
C........	remainder	7.0 ±0.8	1.3	2.0 ±0.8	0.05	0.3	2.0	...	....	1.0[a]
CC.......	remainder	7.7 ±0.5	1.0±0.2	1.25±0.25	0.05	0.3	0.2	...	....	0.3
F........	remainder	$10.0\begin{cases}+0.7\\-0.5\end{cases}$	1.3	...	$0.25\begin{cases}+0.10\\-0.05\end{cases}$	0.25	0.2	...	....	0.3[b]
G........	remainder	4.5 ±0.5	0.8	$0.8\begin{cases}+0.4\\-0.3\end{cases}$	0.03	0.05	0.05	0.2	....	0.05 each
GG.......	remainder	4.5 ±0.5	0.8	$0.8\begin{cases}+0.4\\-0.3\end{cases}$	0.3	0.3	0.2	0.2	....	0.2
H........	remainder	4.0 ±0.4	0.8	0.6	$1.5\begin{cases}+0.3\\-0.2\end{cases}$	0.2	0.1	...	2.0±0.3	0.2
J........	remainder	0.3	0.6	$5.0\begin{cases}+0.8\\-0.2\end{cases}$	0.05	0.3	0.2	0.2	....	0.3
JJ.......	remainder	0.1	0.6	$5.0\begin{cases}+0.8\\-0.2\end{cases}$	0.05	0.05	0.05	0.2	....	0.05 each
K........	remainder	0.1	0.6	12.5 ±0.5	0.05	0.2	0.1	...	....	0.2
L........	remainder	0.1	0.35	0.25	3.9 ± 0.4	0.6	0.05	0.2	....	0.05 each
M........	remainder	0.15	0.4	7.0 ± 0.5	0.3 ± 0.1	....	0.05	0.2	....	0.05 each
N........	remainder	1.25±0.25	0.4	5.0 ± 0.5	0.5 ± 0.1	0.1	0.05	0.2	....	0.2

[a] Total of all elements other than aluminum not to exceed 13.0 per cent.

[b] Iron plus silicon not more than 1.8 per cent.

Inspection

8. (*a*) Inspection may be made at the manufacturer's works where the alloys are made, or at the point where they are received, at the option of the purchaser.

(*b*) If the purchaser elects to have inspection made at the manufacturer's works, the inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

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[1] A.S.T.M. Methods of Chemical Analyses of Metals, p. 95 (1936).

Rejection

9. Material which does not conform to the requirements of these specifications may be rejected, and if rejected shall be replaced by the manufacturer without charge to the purchaser. The full weight of the rejected material shall be returned to the manufacturer.

Rehearing

10. In the case of dissatisfaction regarding rejections based on quality or chemical composition requirements specified in Sections 3 and 4, the manufacturer may make claim for a rehearing as the basis of arbitration within five working days of receipt by the manufacturer of the rejection notification.

EXPLANATORY NOTE

NOTE.—Physical properties and explanatory notes pertaining to the alloys covered by these specifications when used in the form of sand castings are given in the Tentative Specifications for Aluminum-Base Alloy Sand Castings (A.S.T.M Designation: B 26 - 37 T) of the American Society for Testing Materials.[1]

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[1] See p. 678.

TENTATIVE SPECIFICATIONS

FOR

ALUMINUM-BASE ALLOYS IN INGOT FORM FOR PERMANENT MOLD CASTINGS[1]

A.S.T.M. Designation: B 112 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

Scope

1. These specifications cover commercial aluminum-base alloys in ingot form for remelting for the manufacture of permanent mold castings. The specific gravity of these alloys does not exceed 3.0. Thirteen alloys are specified and are designated Alloys 1, 1A, 2, 3, 4, 5, 6, 7, 8, 9, 9A, 10, and 11 (see Explanatory Note).

Process

2. The alloys may be made by any approved process.

Quality

3. The material covered by these specifications shall be of uniform quality and shall be free from dross, slag, and other harmful contamination.

Chemical Composition

4. The alloy shall conform to the chemical composition requirements specified in Table I.

Sampling for Chemical Analysis

5. (*a*) The sample for determining the chemical composition shall be taken by sawing, drilling, or milling the sample ingot in such a manner as to be representative of its average cross-section.

(*b*) The saw, drill, or cutter used for taking the sample shall be thoroughly cleaned. No lubricant shall be used in the operation, and the sawings or metal chips shall be carefully treated with a magnet to remove any particles of iron introduced in taking the sample.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee B-7 on Light Metals and Alloys, Cast and Wrought.

Methods of Chemical Analysis

6. The chemical analysis shall be made in accordance with the Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys (A.S.T.M. Designation: B 40 – 36 T) of the American Society for Testing Materials.[1]

Number of Tests

7. If the ingots are shipped in carload lots of the same alloy, not less than five ingots shall be taken at random from the carload for sampling.

TABLE I.—CHEMICAL COMPOSITIONS OF ALUMINUM-BASE ALLOY INGOTS FOR PERMANENT MOLD CASTINGS.

NOTE 1.—Where single limits are shown in this table these indicate the maximum amount permitted.

NOTE 2.—Analysis shall regularly be made only for the elements specifically mentioned in this table. If, however, the presence of other elements is indicated in the course of routine analysis, further analysis shall be made to determine that the total of these other elements is not present in excess of the limits specified in the last column of the table.

NOTE 3.—The following applies to all specified limits in this table: For purposes of acceptance and rejection, an observed value or a calculated value obtained from analysis should be rounded off to the nearest unit in the last right-hand place of figures used in expressing the specified limit.

NOTE 4.—Titanium is permissible in Alloys 3, 4, 9, 9A, and 10, but the maximum shall not exceed 0.2 per cent.

Alloy	Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Magnesium, per cent	Nickel, per cent	Zinc, per cent	Manganese, per cent	Total Other Impurities, max., per cent
1	remainder	$4.5\pm\underline{0.4}$	0.8	$2.5\pm\underline{0.4}$	0.2	....	0.2	0.3	0.3
1A	remainder	$4.5\pm\underline{0.4}$	0.8	$2.5\pm\underline{0.4}$	0.05	....	0.2	0.3	0.3
2	remainder	$7.0\pm\underline{0.8}$	1.1	$3.0\pm\underline{0.8}$	0.1	....	2.5	0.3	1.0[a]
3	remainder	$4.5\pm\underline{0.4}$	0.8	$5.5\pm\underline{0.5}$	0.1	....	0.5	0.3	0.5
4	remainder	$10.0\left\{\begin{matrix}+0.7\\-0.5\end{matrix}\right.$	1.3	0.8	$0.25\left\{\begin{matrix}+0.10\\-0.05\end{matrix}\right.$	0.3	0.4	0.25	0.3
5	remainder	$10.1\pm\underline{0.6}$	1.0	$4.0\pm\underline{0.5}$	$0.25\left\{\begin{matrix}+0.10\\-0.05\end{matrix}\right.$	....	0.5	0.3	0.5
6	remainder	$1.0\pm\underline{0.4}$	1.1	$12.0\pm\underline{0.8}$	1.1±0.15	2.5±0.4	0.1	0.05	0.2
7	remainder	$1.5\pm\underline{0.4}$	0.7	12.3±0.6	0.6±0.1	0.05	0.4	0.75±0.15	0.3
8	remainder	0.2	0.6	$11.0\pm\underline{0.8}$	0.05	....	0.3	0.5	0.5
9	remainder	0.3	0.6	$5.0\left\{\begin{matrix}+0.8\\-0.3\end{matrix}\right.$	0.05	....	0.3	0.3	0.3
9A	remainder	0.1	0.6	$5.0\left\{\begin{matrix}+0.8\\-0.3\end{matrix}\right.$	0.05	....	0.05	0.05	0.05 each
10	remainder	0.15	0.4	$7.0\pm\underline{0.5}$	0.3±0.1	....	0.05	0.05	0.05 each
11	remainder	$4.0\pm\underline{0.4}$	0.8	0.6	$1.5\left\{\begin{matrix}+0.3\\-0.2\end{matrix}\right.$	2.0±0.3	0.1	0.2	0.2

[a] Total of all elements other than aluminum not to exceed 14.0 per cent

If the shipment is in less than carload lots, one sample ingot shall be taken for each 6000 lb. (2721.6 kg.) or fraction thereof. When it is deemed necessary, a sample may be taken from each melt of 500 lb. (226.8 kg.) or more of the alloy.

Inspection

8. (*a*) Inspection may be made at the manufacturer's works where the alloys are made, or at the point where they are received, at the option of the purchaser.

(*b*) If the purchaser elects to have inspection made at the manufacturer's works, the inspector representing the purchaser shall have free entry, at all times while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the material ordered. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

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[1] A.S.T.M. Methods of Chemical Analyses of Metals, p. 95 (1936).

Rejection

9. Material which does not conform to the requirements of these specifications may be rejected, and if rejected shall be replaced by the manufacturer without charge to the purchaser. The full weight of the rejected material shall be returned to the manufacturer.

Rehearing

10. In the case of dissatisfaction regarding rejections based on quality or chemical composition requirements specified in Sections 3 and 4, the manufacturer may make claim for a rehearing as the basis of arbitration within five working days of receipt by the manufacturer of the rejection notification.

EXPLANATORY NOTE

NOTE.—Supplementary data pertaining to the alloys covered by these specifications when used in the form of permanent mold castings are given in the Tentative Specifications for Aluminum-Base Alloy Permanent Mold Castings (A.S.T.M. Designation: B 108 – 37 T) of the American Society for Testing Materials.[1]

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[1] See p. 674.

TENTATIVE SPECIFICATIONS
FOR
ALUMINUM-BASE ALLOY PERMANENT MOLD CASTINGS[1]

A.S.T.M. Designation: B 108 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1936; REVISED, 1937.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

Scope

1. These specifications cover aluminum-base alloy permanent mold castings having a specific gravity of 3 or less. Eleven types of alloys are specified and are designated Alloys 1 to 11, inclusive. (See Table of Supplementary Data in the Appendix.)

2. These specifications refer to castings of two classes: namely,
Standard commercial quality for regular applications, and
Class A alloys where the greatest freedom from impurities is required for special applications.

Manufacture

3. The finished castings shall be uniform in composition and free from blow holes, cracks, shrinks, and other injurious defects.

Chemical Composition

4. The alloys shall conform to the chemical composition requirements specified in Table I.

Sampling for Chemical Analysis

5. (*a*) The samples for chemical analyses shall be selected as far as possible by heats or melts, unless otherwise mutually agreed upon by the manufacturer and the purchaser.

(*b*) The sample for determining the chemical composition shall be taken by sawing, drilling or milling the casting or test specimen in such a manner as to be representative of the material on test.

(*c*) The saw, drill, cutter, or other tool used for sampling the casting shall be thoroughly cleaned. No lubricant shall be used in the operation, and the sawings or metal chips shall be carefully treated with a magnet to remove any particles of iron introduced in taking the sample.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee B-7 on Light Metals and Alloys, Cast and Wrought.

Method of Chemical Analysis

6. The chemical analysis shall be made in accordance with the Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys (A.S.T.M. Designation: B 40 – 36 T) of the American Society for Testing Materials.[1]

Physical Properties

7. The physical properties as determined from test specimens shall conform to the requirements specified in Table II except as noted.

TABLE I.—CHEMICAL COMPOSITIONS OF ALUMINUM-BASE ALLOY PERMANENT MOLD CASTINGS.

NOTE 1.—Where single limits are shown these indicate the maximum amount permitted.

NOTE 2.—Analysis shall regularly be made only for the elements specifically mentioned in this table. If, however, the presence of other elements is indicated in the course of routine analysis, further analysis shall be made to determine that the total of these other elements is not present in excess of limits specified in the last column of the table.

NOTE 3.—The following applies to all specified limits in this table: For purposes of acceptance and rejection, an observed value or a calculated value obtained from analysis should be rounded off to the nearest unit in the last right-hand place of figures used in expressing the specified limit.

NOTE 4.—Titanium is permissible in Alloys 3, 4, 9, 9A and 10, but the maximum shall not exceed 0.2 per cent.

Alloy	Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Magnesium, per cent	Nickel, per cent	Zinc, per cent	Manganese, per cent	Total Other Impurities, max., per cent
1............	remainder	4.5±0.5	1.0	2.5±0.5	0.2	....	0.2	0.3	0.3
1A............	remainder	4.5±0.5	1.0	2.5±0.5	0.05	....	0.2	0.3	0.3
2............	remainder	7.0±1.0	1.4	3.0±1.0	0.1	....	2.5	0.3	1.0[a]
3............	remainder	4.5±0.5	1.0	5.5±0.5	0.1	....	0.5	0.3	0.5
4............	remainder	10.0±0.75	1.5	1.0	0.25±0.10	0.3	0.4	0.3	0.3
5............	remainder	10.0±1.0	1.2	4.0±0.5	0.25±0.10	....	0.5	0.3	0.5
6............	remainder	1.0±0.5	1.3	12.0±1.0	1.0±0.25	2.5±0.5	0.1	0.05	0.2
7............	remainder	1.5±0.5	0.9	12.25±0.75	0.55±0.15	0.05	0.4	0.75±0.15	0.3
8............	remainder	0.3	0.8	11.0±1.0	0.05	....	0.3	0.5	0.5
9............	remainder	0.4	0.8	$5.0\begin{cases}+1.0\\-0.5\end{cases}$	0.05	....	0.3	0.3	0.3
9A............	remainder	0.1	0.8	$5.0\begin{cases}+1.0\\-0.5\end{cases}$	0.05	....	0.05	0.05	0.05 each
10............	remainder	0.2	0.5	7.0±0.5	0.3±0.1	....	0.05	0.05	0.05 each
11............	remainder	4.0±0.5	1.0	0.7	1.5±0.3	2.0±0.3	0.1	0.3	0.3

[a] Total of all elements other than aluminum not to exceed 14.5 per cent.

Test Specimens

8. (*a*) The test specimens shall be separately cast in a permanent mold. The specimens shall have a gage section diameter of 0.505 in. ± 0.010 in., and a gage length of not less than 2¼ in. The radius of fillet shall be not less than ½ in. A larger radius is not objectionable. The gage section diameter may be slightly reduced in the middle to promote breaking in this area.

(*b*) If the castings are to be heat treated, the test specimens representing such castings shall be heat treated with the castings they represent. They shall not be machined prior to test except to adapt the grip ends of the specimen to the jaws of the testing machine in such a manner as to assure axial loading.

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[1] A.S.T.M. Methods of Chemical Analyses of Metals, p. 95 (1936).

Tension Tests

9. The tensile properties shall be determined in accordance with the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials.[1]

Number of Tests

10. (*a*) The test specimens shall, so far as possible, be selected by heats or melts but, unless otherwise agreed upon by the purchaser or his representative and the manufacturer, two tension tests shall be made for each unit of 1000 lb. or fraction thereof, of clean castings.

TABLE II.—PHYSICAL PROPERTIES OF ALUMINUM-BASE ALLOY PERMANENT MOLD CASTINGS.

NOTE.—See Table of Supplementary Data in the Appendix.

ALLOY	CONDITION	TENSILE STRENGTH, MIN., LB. PER SQ. IN (TO THE NEAREST 1000 LB. PER SQ. IN.)	ELONGATION IN 2 IN., MIN., PER CENT (TO THE NEAREST 1 PER CENT)
1	Heat Treatment No. 1	33 000	4.5
	Heat Treatment No. 2	42 000	1[a]
1A	Heat Treatment No. 1	33 000	4.5
	Heat Treatment No. 2	42 000	1[a]
2	As-cast	23 000	....
3	As-cast	23 000	....
4	Heat Treatment No. 1	28 000	....
	Heat Treatment No. 2	38 000	....
5	As-cast	26 000	....
6	Heat Treatment No. 1	28 000	....
	Heat Treatment No. 2	36 000	....
7	Heat Treatment No. 1	32 000	....
	Heat Treatment No. 2	36 000	....
8	As-cast	27 000	3
9	As-cast	21 000	2
9A	As-cast	21 000	2.5
10	Heat Treatment No. 1	28 000	5
	Heat Treatment No. 2	30 000	3
11	Heat Treatment No. 1	33 000	....
	Heat Treatment No. 2	40 000	....

[a] For information only.

(*b*) If any test specimen shows defective machining or flaws, it may be discarded; in which case the purchaser or his representative and the manufacturer shall agree upon the selection of another specimen in its stead.

Retests

11. If the results of the tension tests do not conform to the tensile properties specified in Section 7, the castings may be reheat-treated but not more than twice. The results of acceptable tests shall conform to the tensile property requirements as specified in Section 7.

Inspection

12. (*a*) Inspection may be made at the manufacturer's works where the castings are made, or at the point at which they are received, at the option of the purchaser.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 833.

(*b*) The general requirements specified in Section 3 shall be checked by visual inspection of the castings or by comparison with observational standards where such standards are established. It is advisable that observational standards representing acceptable material be set up which will govern when any question arises as to what defects are allowable under the best commercial practice.

(*c*) If the purchaser elects to have inspection made at the manufacturer's works, the manufacturer shall afford the inspector representing the purchaser all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

Rejection

13. Castings which show injurious defects revealed by machining operations subsequent to acceptance may be rejected, and if rejected shall be replaced by the manufacturer without charge to the purchaser. The full weight of the original material rejected shall be returned to the manufacturer.

Rehearing

14. In the case of dissatisfaction regarding rejections based on chemical composition and tensile property requirements as specified in Sections 4 and 7, respectively, the manufacturer may make claim for a rehearing as a basis of arbitration within a reasonable time after receipt by the manufacturer of the rejection notification.

APPENDIX

The data in the following table are approximate and are supplied for general information only:

TABLE OF SUPPLEMENTARY DATA

Alloy	Approximate Specific Gravity	Corrosion Resistance	Casting Properties	Response to Heat Treatment	Principal Use	Machinability
1	2.76	Good	Good	Yes	General, where high strength and fair ductility are required	Good
1A	2.76	Good	Good	Yes	General, where high strength and high ductility are required.	Good
2	2.83	Fair	Good	Yes	General, automotive cylinder heads and washing machine agitators	Excellent
3	2.79	Fair	Excellent	No	General	Good
4	2.90	Fair	Good	Yes	Automotive pistons	Excellent
5	2.88	Fair	Good	Yes	General	Good
6	2.68	Good	Fair	Yes	Pistons	Fair
7	2.67	Good	Fair	Yes	Pistons	Fair
8	2.64	Excellent	Excellent	No	General thin sections	Poor
9	2.66	Good	Good	No	General and cooking utensils	Poor
9A	2.66	Excellent	Good	No	General with maximum corrosion resistance.	Poor
10	2.70	Excellent	Excellent	Yes	General where high strength and corrosion resistance are required	Good
11	2.78	Good	Fair	Yes	Aircraft and Diesel engine pistons	Good

TENTATIVE SPECIFICATIONS
FOR
ALUMINUM-BASE ALLOY SAND CASTINGS[1]

A.S.T.M. Designation: B 26 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1918; ADOPTED IN AMENDED FORM, 1921; REVISED, 1931;
REISSUED AS TENTATIVE, 1933; REVISED, 1936, 1937.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

Scope

1. These specifications cover aluminum-base alloy sand castings having a specific gravity of 3 or less. Twelve alloys are specified and are designated Alloys C, CC, F, G, GG, H, J, JJ, K, L, M, and N (see Explanatory Notes).

Manufacture

2. The finished casting shall be uniform in composition and as free from blow holes, cracks, shrinks, and other injurious defects as the best commercial practice will permit.

3. The responsibility of furnishing castings that can be laid out and machined to the finished dimensions within the specified tolerances as shown on the blueprints or drawings shall rest with the manufacturer, except where pattern equipment is furnished by the purchaser. Sufficient stock shall be allowed for shrinkage and, where required, for finishing; but castings of excessive size or weight shall not be furnished.

Chemical Composition

4. The alloys shall conform to the chemical composition requirements specified in Table I.

Sampling for Chemical Analysis

5. (*a*) Samples for chemical analyses shall be selected as far as possible by heats or melts, unless otherwise mutually agreed upon by the manufacturer and the purchaser.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee B-7 on Light Metals and Alloys, Cast and Wrought.

These specifications are in effect a revision of and replace the former Standard Specifications for Aluminum-Base Alloy Sand Castings (A.S.T.M. Designation: B 26 - 31), which standard was accordingly discontinued in 1933.

(*b*) The sample for determining the chemical composition shall be taken by sawing, drilling or milling the casting or test specimen in such a manner as to be representative of the material on test.

(*c*) The saw, drill, cutter or other tool used for sampling the casting shall be thoroughly cleaned. No lubricant shall be used in the operation, and the sawings or metal chips shall be carefully treated with a magnet to remove any particles of iron introduced in taking the sample.

TABLE I.—CHEMICAL COMPOSITIONS OF ALUMINUM-BASE ALLOY SAND CASTINGS.

NOTE 1.—Where single limits are shown, these indicate the maximum amount permitted.

NOTE 2.—Analysis shall regularly be made only for the elements specifically mentioned in this table. If, however, the presence of other elements is indicated in the course of routine analysis, further analysis shall be made to determine that the total of these other elements is not present in excess of the limits specified in the last column of the table

NOTE 3.—The following applies to all specified limits in this table: For purposes of acceptance and rejection, an observed value or a calculated value obtained from analysis should be rounded off to the nearest unit in the last right-hand place of figures used in expressing the specified limit.

Alloy	Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Magnesium, per cent	Manganese, per cent	Zinc, per cent	Titanium, per cent	Nickel, per cent	Total Other Impurities, max., per cent
C..........	remainder	7.0±1.0	1.5	2.0±1.0	0.05	0.3	2.2	....	....	1.0[a]
CC.........	remainder	$7.7\left\{\begin{matrix}+0.8\\-0.7\end{matrix}\right.$	$1.0\left\{\begin{matrix}+0.4\\-0.2\end{matrix}\right.$	1.25±0.25	0.05	0.3	0.2	....	....	0.3
F..........	remainder	10.0±0.75	1.5	....	0.25±0.10	0.3	0.2	....	....	0.3[b]
G..........	remainder	4.5±0.5	1.0	1.2	0.03	0.05	0.05	0.2	....	0.05 each
GG.........	remainder	4.5±0.5	1.2	1.2	0.3	0.3	0.2	0.2	....	0.2
H..........	remainder	4.0±0.5	1.0	0.7	1.5±0.3	0.3	0.1	....	2.0±0.3	0.3
J..........	remainder	0.4	0.8	$5.0\left\{\begin{matrix}+1.0\\-0.5\end{matrix}\right.$	0.05	0.3	0.2	0.2	....	0.3
JJ.........	remainder	0.1	0.8	$5.0\left\{\begin{matrix}+1.0\\-0.5\end{matrix}\right.$	0.05	0.05	0.05	0.2	....	0.05 each
K..........	remainder	0.15	0.8	12.5±0.5	0.05	0.2	0.1	....	....	0.2
L..........	remainder	0.1	0.4	0.25	3.75±0.50	0.6	0.05	0.2	....	0.05 each
M..........	remainder	0.2	0.5	7.0±0.5	0.3±0.1	....	0.05	0.2	....	0.05 each
N..........	remainder	1.25±0.25	0.5	5.0±0.5	0.5±0.1	0.1	0.05	0.2	....	0.2

[a] Total of all elements other than aluminum not to exceed 13.5 per cent.

[b] Iron plus silicon not more than 2.0 per cent.

Method of Chemical Analysis

6. The chemical analysis shall be made in accordance with the Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys (A.S.T.M. Designation: B 40 - 36 T) of the American Society for Testing Materials.[1]

Physical Properties

7. The physical properties as determined from test specimens shall conform to the requirements specified in Table II.

Test Specimens

8. (*a*) The test specimens shall be separately cast in green sand and shall be cast to size according to the dimensions shown in Fig. 1.

(*b*) If the castings are to be heat treated, the test specimens representing such castings shall be heat treated with the castings they represent. They shall not be machined prior to test except to adapt the grip ends of the specimen to the jaws of the testing machine in such a manner as to assure axial loading.

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[1] A.S.T.M. Methods of Chemical Analyses of Metals, p. 95 (1936).

Tension Tests

9. The tensile properties shall be determined in accordance with the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials.[1]

Number of Tests

10. (*a*) The test specimens shall, so far as possible, be selected by heats or melts but, unless otherwise agreed upon by the purchaser or his representative and the manufacturer, two tension tests shall be made for each unit of 500 lb. or fraction thereof.

TABLE II.—PHYSICAL PROPERTIES OF ALUMINUM-BASE ALLOY SAND CASTINGS.

NOTE.—See Explanatory Notes.

ALLOY	CONDITION	TENSILE STRENGTH, MIN., LB. PER SQ. IN. (TO THE NEAREST 1000 LB. PER SQ. IN.)	ELONGATION IN 2 IN., MIN., PER CENT (TO THE NEAREST 1 PER CENT)
C	As-cast	19 000	1[a]
CC	As-cast	19 000	3
F	As-cast	19 000	[b]
G	Heat Treatment No. 1	29 000	6
	Heat Treatment No. 2	32 000	3
	Heat Treatment No. 3	36 000	[b]
GG	Heat Treated	36 000	[b]
H	Heat Treated	32 000	[b]
J	As-cast	17 000	3
JJ	As-cast	17 000	3
K	As-cast	24 000	5
L	As-cast	22 000	6
M	Heat Treatment No. 1	26 000	5
	Heat Treatment No. 2	30 000	3
N	Heat Treatment No. 1	27 000	4
	Heat Treatment No. 2	32 000	2
	Heat Treatment No. 3	25 000	1[a]

[a] For information only.
[b] Not required.

(*b*) If any test specimen shows defective machining or flaws, it may be discarded; in which case the purchaser or his representative and the manufacturer shall agree upon the selection of another specimen in its stead.

Retests

11. If the results of the tension tests do not conform to the tensile properties specified in Section 9, the castings may be reheat-treated but not more than twice. The results of acceptable tests shall conform to the tensile property requirements as specified in Section 9.

Inspection

12. (*a*) Inspection may be made at the manufacturer's works where the castings are made, or at the point at which they are received, at the option of the purchaser.

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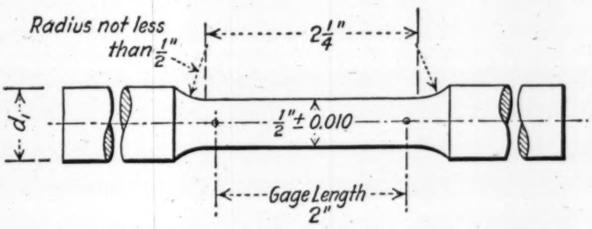
[1] 1936 Book of A.S.T.M. Standards, Part I, p. 833.

(*b*) The general requirements specified in Section 3 shall be checked by visual inspection of the castings or by comparison with observational standards where such standards are established. It is advisable that observational standards representing acceptable material be set up which will govern when any question arises as to what defects are allowable under the best commercial practice.

(*c*) If the purchaser elects to have inspection made at the manufacturer's works, the manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.



NOTE:

The Gage Length, Parallel Section and Fillets shall be as shown, but the Ends may be of any Shape to fit the Holders of the Testing Machine in such a way, that the Load shall be Axial.

In Order to promote the Breaking of the Specimen in the Center it is permissible to use a Taper in the Reduced Section from the Fillets to the Center of not to exceed 0.010 in.

It is recommended, that Diameter d_1 be from $\frac{3}{4}$" to 1."

FIG. 1.—Tension Test Specimen.

Rejection

13. Castings which show injurious defects revealed by machining operations subsequent to acceptance may be rejected, and if rejected shall be replaced by the manufacturer without charge to the purchaser. The full weight of the original material rejected shall be returned to the manufacturer.

Rehearing

14. In the case of dissatisfaction regarding rejections based on chemical composition and tensile property requirements specified in Sections 6 and 9, respectively, the manufacturer may make claim for a rehearing as the basis of arbitration within five working days of receipt by the manufacturer of the rejection notification.

EXPLANATORY NOTES

Alloy C.—The specific gravity of Alloy C is about 2.83 to 2.86. The pattern shrinkage is about 0.156 ($\frac{5}{32}$) in. per ft. It has somewhat better machining qualities than Alloy CC. It is used for about the same purposes as Alloy CC, particularly for automotive work, such as crank and transmission cases and similar parts.

Alloy CC.—Alloy CC has a specific gravity of about 2.83. The pattern shrinkage is about 0.156 ($\frac{5}{32}$) in. per ft. It supersedes the 8 per cent copper alloy without silicon as it possesses better casting characteristics, especially in the more difficult work where higher pouring temperatures than those usually employed are found necessary. The tendency towards cracks and shrinks is considerably overcome by the careful control of the iron and silicon contents. This alloy is used for general casting purposes.

Alloy F.—The specific gravity of Alloy F is about 2.90 and the pattern shrinkage about 0.156 ($\frac{5}{32}$) in. per ft. This alloy is used for pistons, cylinder heads, valve guides, and bearings or other parts where hardness and good bearing qualities are essential. Together with Alloy H it possesses the property of retaining at higher temperatures a greater percentage of its room temperature strength than any other alloy in this group. This alloy may be heat treated, in which case substantially higher strength and hardness or other desirable characteristics are obtained.

Alloy G.—Alloy G has a specific gravity of about 2.78. It is used for general casting purposes where a combination of lightness, higher strength and ductility is desired. The various strengths are obtained through different heat treatments. Heat Treatment No. 1 gives maximum elongation and resistance to shock. It is a solution heat treatment and may be specified where the most rugged service is required. On standing at room temperatures, the alloy age hardens over a period of a few months to develop mechanical properties approaching those of Heat Treatment No. 2. Heat Treatment No. 2 is a solution and precipitation heat treatment, and should be specified where a higher initial strength, yield strength and hardness are desired. Heat Treatment No. 3 should be specified where the highest strength and hardness are desired and lower ductility is not objectionable. This alloy with the various heat treatments is used in general for high strength parts in street cars and busses, parts for internal combustion engines, outboard motor parts and other marine castings.

Alloy GG.—Alloy GG is used in the heat-treated condition where high strength and maximum hardness are desired, such as bearing caps.

Alloy H.—The specific gravity of Alloy H is about 2.80. It is usually employed in the heat-treated condition. The alloy is used for pistons, cylinder heads, valve guides and bearings or other parts where hardness or good bearing qualities are essential. Together with Alloy F it possesses the property of retaining at higher temperatures a greater percentage of its room-temperature strength than any other alloy in this group.

Alloy J.—Alloy J is somewhat lighter than aluminum, having a specific gravity of about 2.69. It withstands corrosion better than the copper alloys. The free flowing property of the alloy when molten makes it easier to pour castings of thin section or intricate design. Its leak proof properties are valuable where pressure tightness is essential. It has a lower strength and yield strength than the copper aluminum alloys and poorer machining qualities. It is especially suitable for automobile body parts, cooking utensils, architectural and marine castings.

Alloy JJ.—Alloy JJ is similar to Alloy J except that it is held to closer chemical composition limits which results in greater corrosion resistance.

Alloy K.—The specific gravity of Alloy K is about 2.68. Immediately before pouring, the alloy is treated by a process called "modification." Its corrosion resistance is good, especially to salt water. It has good foundry characteristics and can be used for castings having both thin and heavy sections. It is not as readily machined and has a lower yield strength in proportion to tensile strength than most of the copper-aluminum alloys. It is used for marine castings, bus, street car and engine parts, and for thin castings such as radiators, small motor housings and meter cases.

Alloy L.—Alloy L has the highest resistance to corrosion of any of the commercial aluminum casting alloys. It is not so easily cast as some of the other alloys, which fact should be considered when designing complicated castings. Its specific gravity is approximately 2.63.

Alloy M.—The specific gravity of Alloy M is approximately 2.65. It has casting qualities and resistance to corrosion comparable with those of Alloy J (see Note on Alloy J). Its higher mechanical properties result from heat treatment processes. The alloy in Heat Treatment No. 1 age hardens on standing at room temperatures and after a few months its mechanical properties approach those of Heat Treatment No. 2.

Alloy N.—Alloy N has a specific gravity of approximately 2.68. It has good casting qualities. It is used extensively for liquid cooled cylinder heads and other castings which are subjected to elevated temperatures in service. The alloy in Heat Treatment No. 1 age hardens and develops properties approaching those of Heat Treatment No. 2. Heat Treatment No. 3 is applied principally to castings which are to be used at high temperatures and serves to stabilize the dimensions under those conditions.

TENTATIVE SPECIFICATIONS

FOR

COPPER AND COPPER-ALLOY SEAMLESS CONDENSER TUBES AND FERRULE STOCK[1]

A.S.T.M. Designation: B 111 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

Scope

1. These specifications cover seamless tubes and ferrule stock of various copper alloys, for use in surface condensers, evaporators, and heat exchangers.

2. The tubes shall be cold worked to size from an alloy made from lake or electrolytic copper in combination with one or more of the following elements conforming to the requirements of standard specifications of the American Society for Testing Materials, as follows:

(*a*) *Lake or Electrolytic Copper.*—Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars (A.S.T.M. Designation: B 4)[2] or for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars (A.S.T.M. Designation: B 5).[3]

(*b*) *Zinc, No. 1 High Grade.*—Standard Specifications for Slab Zinc (Spelter) (A.S.T.M. Designation: B 6).[4]

(*c*) *Tin.*—"Straits" tin or its equivalent.

(*d*) *Nickel, Electrolytic Grade.*—Standard Specifications for Nickel (A.S.T.M. Designation: B 39).[5]

(*e*) *Aluminum, Grade 99.5.*—Standard Specifications for Aluminum Ingots for Remelting (A.S.T.M. Designation: B 24).[6]

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee B-5 on Copper and Copper Alloys, Cast and Wrought.

These specifications are in effect a revision and consolidation of, and replace the former Standard Specifications for Seamless Admiralty Condenser Tubes and Ferrule Stock (B 44 – 33), and for Seamless Muntz Metal Condenser Tubes and Ferrule Stock (B 56 – 33), and also the former Tentative Specifications for Seamless Copper-Nickel Alloy Condenser Tubes and Ferrule Stock (B 104 – 36 T), which specifications were accordingly discontinued in 1937.

[2] 1936 Book of A.S.T.M. Standards, Part I, p. 526.

[3] *Ibid.*, p. 531.

[4] 1937 Supplement to Book of A.S.T.M. Standards, p. 43.

[5] 1936 Book of A.S.T.M. Standards, Part I, p. 684.

[6] *Ibid.*, p. 524.

(*f*) Scrap shall not be used except such as may result from the manufacture of articles of the same composition.

Manufacture

3. (*a*) Condenser tubes shall be so processed as to enable the tubes to pass the physical and metallographic tests prescribed in these specifications.

(*b*) Tubes for ferrule stock shall be annealed sufficiently to be fully recrystallized, but are not subject to the expanding test specified in Section 7.

Chemical Composition

4. The material shall conform to the following requirements as to chemical composition (see Explanatory Note):

Alloy	Copper,[a] per cent	Tin, per cent	Aluminum, per cent	Nickel, per cent	Lead, max., per cent	Iron, max., per cent	Zinc, per cent	Manganese, per cent	Phosphorus, max., per cent	Arsenic, per cent
Muntz metal[b]	59.0 to 63.0	....	....	....	0.3	0.07	remainder	......	......	....
Admiralty[b]	70.0 min.	0.90 to 1.20	....	....	0.075	0.06	remainder	......	......	....
Red brass	84.0 min.	....	....	....	0.075	0.06	remainder	......	......	....
Aluminum brass[b]	76.0 min.	1.25 max.	1.75 to 2.50	1.25 max.	0.075	0.06	remainder	......	......	....
Aluminum bronze	93.5 min.	....	5.00 min.	....	.....	.....	....	......	......	....
70-30 copper-nickel	remainder	1.50 max.	....	29.0 to 33.0	0.05	0.5	1.0 max.	1.0 max.	......	....
80-20 copper-nickel:										
Type A	remainder	1.0 max.	....	19.0 to 23.0	0.05	0.5	1.0 max.	1.0 max.	......	....
Type B	remainder	1.0 max.	....	19.0 to 23.0	0.05	0.5	3.0 to 6.0	1.0 max.	......	....
Copper	99.90 min.	....	....	....	.....	.....	....	......	0.035	....
Arsenical copper	99.20 min.	....	....	....	.....	.....	....	......	......	0.15 to 0.75

[a] Silver counting as copper.

[b] If agreed upon between the manufacturer and the purchaser, these alloys may contain a maximum of 0.25 per cent antimony or a maximum of 0.1 per cent arsenic.

Sample for Chemical Analysis

5. The sample for chemical analysis shall consist of drillings, millings, or clippings taken from two separate tubes from each lot of 600 tubes or fraction thereof, equal quantities being taken from each tube and thoroughly mixed. Samples so prepared shall be divided into three equal parts, each of which shall be placed in a sealed package, one for the manufacturer, one for the purchaser, and one for an umpire, if necessary.

Microscopic Examination

6. (*a*) A microscopic examination of the annealed tubes selected for test, except those of Muntz metal, shall show sufficient annealing for complete recrystallization with a uniform grain size not in excess of 0.045 mm. in average diameter. Muntz metal tubes shall have been so annealed that when magnified at 75 diameters a properly prepared specimen shall show uniform and complete recrystallization.

(*b*) The test specimen for the microscopic examination shall be a radial longitudinal section of the tube made by cutting along a plane containing the longitudinal axis of the tube (*c*) The determination of grain size shall be made by comparison of micrographs representing the specimens selected for test with micrographs of standard specimens for which the grain size has been accurately determined. The magnification in all such micrographs shall be 75 diameters. The standard micrographs shall be taken from specimens, the grain size of which has been determined by Jeffries' method for grain size measurements, as described in the Standard Rules Governing the Preparation of Micrographs of Metals and Alloys, Including Recommended Practice for Photography as Applied to Metallography (A.S.T.M. Designation: E 2) of the American Society for Testing Materials,[1] at a magnification of 250 diameters. Reproductions of micrographs so prepared are shown in Plate II in the Standard Rules E 2.[1]

Expanding Test

7. (*a*) The annealed tubes selected for test shall be capable of standing the following test without showing signs of cracking or rupture: A pin having a taper of $1\frac{1}{2}$ in. per ft. shall be driven into the tube until the inside diameter at one end has been increased 20 per cent. The foregoing applies to all alloys except Muntz metal and copper nickel, which shall be required to withstand an expansion of 15.0 and 30.0 per cent, respectively.

(*b*) Hard-drawn tubes other than copper and arsenical copper are not subject to this test unless specified end-annealed.

Strain Test

8. A test specimen 6 in. in length shall stand, without cracking, an immersion for 15 min. in an aqueous mercurous-nitrate solution containing 100 g. of mercurous nitrate and 13 ml. of HNO_3 (sp. gr. 1.42) per liter of solution. In the case of hard-drawn tubes, end-annealed, the test specimen shall be cut from the central unannealed portion of the tube.

Hydrostatic Test

9. Each tube shall stand an internal hydrostatic pressure of 1000 lb. per sq. in., without leakage.

Number of Tests

10. Tubes shall be grouped into lots of 600 tubes or fraction thereof. Two tubes shall be selected from each lot and subjected to the physical tests specified in Sections 6 to 8, inclusive.

Retests

11. If any test specimen representing a lot fails to meet the requirements of Sections 4, 6 and 7, two additional specimens, at the option of the manufacturer, may be taken as before, and submitted for check analysis or subjected to any tests in which the original specimen failed, but each of these specimens shall conform to the requirements specified.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 763.

Permissible Variations in Dimensions and Weight

12. (*a*) *Diameter*.—The outside diameter of the tubes shall not vary from that specified by more than the following amounts, as measured by "go" and "no go" ring gages:

NOMINAL DIAMETER, IN.	PERMISSIBLE VARIATION,[a] IN.
Up to 0.500, incl	±0.002
Over 0.500 to 0.740, incl	±0.0025
Over 0.740 to 1.000, incl	±0.003
Over 1.000 to 1.250, incl	±0.0035
Over 1.250 to 1.500, incl	±0.004

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[a] For copper-nickel tubes the permissible variation in each size class shall be ±0.0005 in. larger than the tolerances in the above table.

(*b*) *Thickness*.—No tube at its thinnest point shall be less than the thickness specified.

(*c*) *Length*.—The length shall be not less than that ordered, when measured at a temperature of 20 C. (68 F.), but may be more than that ordered by the amounts in the following table:

ORDERED LENGTH, FT.	PERMISSIBLE VARIATION, IN.
Up to 15, incl	$+\frac{1}{16}$
Over 15 to 20, incl	$+\frac{3}{32}$
Over 20	$+\frac{1}{8}$

(*d*) *Weight*.—No lot of tubes shall exceed the calculated weight by more than 5.0 per cent. One cubic inch of the various alloys covered by these specifications shall be assumed to weigh:

ALLOY		UNIT WEIGHT, LB. PER CU. IN.
Muntz metal		0.303
Admiralty		0.308
Red brass		0.316
Aluminum brass		0.301
Aluminum bronze		0.295
70–30 copper-nickel		0.323
80–20 copper-nickel	Type A	0.323
	Type B	0.320
Copper		0.323
Arsenical copper		0.323

Workmanship

13. The tubes shall be round, straight, and uniform in thickness throughout. They shall be free from cracks, seams, slivers, scale, and other surface defects, both inside and outside.

Finish

14. All tubes shall be bright annealed or acid cleaned after the final annealing, unless otherwise specified.

Inspection

15. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the tubes are being furnished in accordance with these specifications. All tests (except check analyses)

and inspection shall be made at the place of manufacture, prior to shipment, unless otherwise specified, and shall be so conducted as not to interfere unnecessarily with the operation of the works.

EXPLANATORY NOTE

NOTE.—Individual manufacturers of condenser tubes offer modifications of the types of alloys covered by these specifications. Some of these are proprietary alloys and many are sold under a trade-mark. These specifications may be considered as covering these special alloys when the change in analysis is specifically agreed upon by both the manufacturer and the purchaser, or when the purchaser waives adherence to stipulations of Section 4.

TENTATIVE SPECIFICATIONS
FOR
COPPER-SILICON ALLOY WIRE FOR GENERAL PURPOSES[1]

A.S.T.M. Designation: B 99 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1935; Revised, 1935, 1936, 1937.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

Scope

1. These specifications cover round copper-silicon alloy wire for general structural purposes except for electrical transmission cable.

Manufacture

Manufacture

2. The material shall be copper-silicon alloy of such quality and purity that when finished, it shall have the properties and characteristics herein specified.

Chemical Properties and Tests

Chemical Composition

3. (*a*) The material shall conform to the following requirements as to chemical composition:

	Type A	Type B	Type C	Type D
Copper, min., per cent	94.80	95.00	90.00	90.00
Silicon, per cent	2.70 to 3.75	0.70 to 1.75	2.30 to 3.75	1.00 to 3.75
Lead, per cent	.....	.....	.....	0.20 to 0.60[a]
One or more of the following elements may be present within the limits specified below:				
Manganese, max., per cent	1.50	0.75	1.50	1.50
Zinc, max., per cent	1.75	1.25	4.25	4.25
Iron, max., per cent	1.60	0.80	1.60	1.60
Tin, max., per cent	0.75	2.25	1.50	2.25
Aluminum, max., per cent	....	2.00	2.00	....
Lead, max., per cent	0.05	0.05	0.05	....
Sum of above elements, min. per cent	99.50	99.50	99.50	99.50

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[a] Either tellurium or selenium may be substituted in Type D alloy in place of lead and in the same amount as that specified for lead.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee B-5 on Copper and Copper Alloys, Cast and Wrought.

Chemical Analysis

4. (*a*) Chemical analysis shall be made on each lot of 5000 lb. or fraction thereof.

(*b*) The sample for chemical analysis shall consist of clean drillings, millings or clippings taken from representative specimens of wire selected for purpose of tests. Equal quantities from each of the specimens sampled shall be thoroughly mixed. Samples so prepared shall be divided into three equal parts, one for the manufacturer, one for the purchaser, and one for an umpire, if necessary.

PHYSICAL PROPERTIES AND TESTS

Physical Properties

5. (*a*) The material shall conform to the requirements as to physical properties shown in Table I.

(*b*) Tests upon a specimen of wire containing a braze or weld shall show at least 95 per cent of the tensile strength of the unbrazed wire. Bend tests shall not be made upon specimens including brazes or welds.

Bend Test

6. All wires of Types A, B, and C alloys shall stand wrapping 360 deg. around a wire of the same diameter without sign of failure. This test shall not be required of material of Type D.

Grain Size Determination

7. In determining grain size, reference should be made to the Standard Rules Governing the Preparation of Micrographs of Metals and Alloys, Including Recommended Practice for Photography as Applied to Metallography (A.S.T.M. Designation: E 2) of the American Society for Testing Materials.[1]

Number of Tests

8. (*a*) Two tension test specimens and one specimen for the bend test or grain size determination, where required, shall be selected from each lot of 10,000 lb. or fraction thereof. Tension and bend tests shall be made on full sections of the wire.

(*b*) If upon testing a specimen from any coil of wire the results are found to be below the values specified in Sections 5 and 6, tests upon two additional specimens shall be made and the average of the three tests shall determine the acceptance or rejection of the coil.

DIMENSIONS AND PERMISSIBLE VARIATIONS

Dimensions and Permissible Variations

9. (*a*) The size shall be expressed as the diameter of the wire in decimal fractions of an inch, using not more than three places of decimals; that is, in mils.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 763.

TABLE I.—PHYSICAL PROPERTIES OF COPPER-SILICON ALLOY WIRE.

TEMPER	PHYSICAL PROPERTIES	UP TO ¼ IN., INCL., IN DIAMETER	OVER ¼ IN. IN DIAMETER
	TYPES A, C AND D ALLOY WIRE		
Annealed	Grain size, max., mm	0.040[a]	0.040[a]
	Tensile strength, lb. per sq. in.	55 000 to 70 000	55 000 to 65 000
	Elongation[b] in 2 in., min., per cent	40	47
	Elongation[b] in 10 in., min., per cent	30	[c]
Eighth-hard	Tensile strength, lb. per sq. in.	62 000 to 78 000	62 000 to 78 000
	Elongation[b] in 2 in., min., per cent	20	28
	Elongation[b] in 10 in., min., per cent	7	[c]
Quarter-hard	Tensile strength, lb. per sq. in.	72 000 to 85 000	72 000 to 85 000
	Elongation[b] in 2 in., min., per cent	15	18
	Elongation[b] in 10 in., min., per cent	5	[c]
Half-hard	Tensile strength, min., lb. per sq. in.	90 000	90 000
	Elongation[b] in 2 in., min., per cent	4	10
	Elongation[b] in 10 in., min., per cent	1.5	[c]
Hard	Tensile strength, min., lb. per sq. in.	115 000	115 000
	Elongation[b] in 2 in., min., per cent	2	6
	Elongation[b] in 10 in., min., per cent	0.75	[c]
Spring[d]	Tensile strength, min., lb. per sq. in.	135 000	130 000
	Elongation[b] in 2 in., min., per cent	1.5	4
	Elongation[b] in 10 in., min., per cent	0.5	[c]
	TYPE B ALLOY WIRE		
Annealed	Grain size, max., mm	0.040[a]	0.040[a]
	Tensile strength, lb. per sq. in.	43 000 to 50 000	43 000 to 50 000
	Elongation[b] in 2 in., min., per cent	33	40
	Elongation[b] in 10 in., min., per cent	25	[c]
Eighth-hard	Tensile strength, lb. per sq. in.	50 000 to 60 000	50 000 to 60 000
	Elongation[b] in 2 in., min., per cent	12	20
	Elongation[b] in 10 in., min., per cent	4	[c]
Quarter-hard	Tensile strength, lb. per sq. in.	60 000 to 75 000	60 000 to 75 000
	Elongation[b] in 2 in., min., per cent	8	15
	Elongation[b] in 10 in., min., per cent	2.5	[c]
Half-hard	Tensile strength, min., lb. per sq. in.	75 000	75 000
	Elongation[b] in 2 in., min., per cent	4	10
	Elongation[b] in 10 in., min., per cent	1	[c]
Hard	Tensile strength, min. lb. per sq. in.	90 000	90 000
	Elongation[b] in 2 in., min., per cent	3	8
	Elongation[b] in 10 in., min., per cent	0.75	[c]
Spring[d]	Tensile strength, min., lb. per sq. in.	100 000	100 000
	Elongation[b] in 2 in., min., per cent	2	6
	Elongation[b] in 10 in., min., per cent	0.50	[c]

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[a] No minimum grain size requirement is specified but all annealed material shall be fully recrystallized.

[b] Elongation may be determined in either the 2-in. or 10-in. gage length but not in both in any instance.

[c] On wire over ¼ in. in diameter the percentage of elongation shall be taken only in 2 in.

[d] The term "Spring Temper" does not necessarily indicate a reduction of eight B. & S. gage numbers on these alloy wires as in the case of brass.

(*b*) Wire shall be accurate in diameter; permissible variations from the nominal diameter shall be within the following requirements:

WIRE DIAMETER	PERMISSIBLE VARIATION OVER	PERMISSIBLE VARIATION UNDER
Under 0.010 in.	0.001 in.	0.001 in.
0.010 in. and over	3 per cent	1 per cent

(*c*) Each coil shall be gaged at three places, one near each end and one approximately at the middle; the coil may be rejected if the measurements at two points being within the accepted limits, the measurement at the third point is not within the accepted limits.

WORKMANSHIP AND FINISH

Workmanship and Finish

10. (*a*) The wire shall be free from all imperfections not consistent with the best commercial practice.

(*b*) Necessary brazes in wire shall be made in accordance with the best commercial practice.

PACKING

Packing

11. (*a*) Package sizes for wire shall be agreed upon by the manufacturer and purchaser in the placing of individual orders.

(*b*) The wire shall be protected against damage in ordinary handling and shipping.

INSPECTION AND REJECTION

Inspection

12. The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications.

Rejection

13. Material which fails to conform to these specifications will be rejected and the manufacturer shall be notified.

TENTATIVE SPECIFICATIONS

FOR

HARD-DRAWN COPPER ALLOY WIRES FOR ELECTRICAL CONDUCTORS[1]

A.S.T.M. Designation: B 105 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1936; Revised, 1937.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

Scope

1. These specifications cover hard-drawn round copper alloy wires for electrical conductors.

Material

2. The material used shall be copper alloy of such nature and composition as to secure by proper treatment the qualities hereinafter specified for the finished wire. The copper used shall be electrolytic or low-resistance lake copper conforming in quality and purity to the requirements of either the Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (A.S.T.M. Designation: B 5)[2] or of the Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs Billets, Ingots, and Ingot Bars (A.S.T.M. Designation: B 4)[3] of the American Society for Testing Materials.

Grades

3. The copper alloy wires may be made in any one of ten distinct grades. The alloys have been designated as Grades Nos. 9 to 85, in accordance with their increasing conductivity, as follows:

Grade No. 9
Grade No. 13
Grade No. 15
Grade No. 20
Grade No. 30
Grade No. 40

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee B-1 on Copper and Copper Alloy Wires for Electrical Conductors.

[2] 1936 Book of A.S.T.M. Standards, Part I, p. 531.

[3] *Ibid.*, p. 526.

Grade No. 55
Grade No. 65
Grade No. 80
Grade No. 85

Chemical Composition

4. (*a*) The copper alloy wires shall conform to the following requirements as to chemical composition. The values given below embrace limits of composition of the different alloys which may be supplied (see Explanatory Note):

Silicon, max., per cent	3.00
Manganese, max., per cent	0.75
Tin, max., per cent	5.00
Aluminum, max., per cent	3.50
Cadmium, max., per cent	1.50
Iron, max., per cent	0.75
Copper, min., per cent	94.00
Sum of the above elements, min., per cent	99.50

(*b*) The maximum percentage of the various alloying elements to be found in any one of the ten grades is stated in the table in Paragraph (*a*). If the purchaser elects to check the composition of any material supplied to meet the performance requirements of any one of the above ten grades, the composition limits should be made the subject of a definite agreement between the manufacturer and purchaser in the placing of each individual order.

Chemical Analyses

5. Analyses may be made on each lot of 5000 lb. or fraction thereof. Millings or clippings shall be made from at least ten separate coils. Equal quantities shall be taken from each coil and shall be thoroughly mixed together. Samples so prepared shall be divided into three equal parts, each of which shall be placed in a sealed package, one for the manufacturer, one for the purchaser, and one for an umpire, if necessary.

Joints

6. Welds and brazes may only be made in rods or in wires prior to final drawing.

Tensile Strength and Elongation

7. (*a*) The alloy wire of a designated grade shall be so drawn that its tensile strength and elongation shall be at least equal to the values given in Table I. Tension tests shall be made upon fair samples. The elongation of wire larger than 0.204 in. in diameter shall be determined as the permanent increase in length, due to the breaking of the wire in tension, measured between bench marks placed upon the wire originally 10 in. apart. The elongation of wire 0.204 in. or smaller in diameter shall be determined by measurements made between the jaws of the testing machine. The zero length shall be the distance between the jaws when

a load equal to 10 per cent of the specified tensile strength shall have been applied, and the final length shall be the distance between the jaws at the time of rupture. The zero length shall be as near 60 in. as possible. The fracture shall be between the bench marks in the case of wire larger than 0.204 in. in diameter and between the jaws in the case of smaller wire, but not closer than 1 in. to either bench mark or jaw.

(*b*) If upon testing a sample from any coil of wire the results are found to be below the values stated in Table I, tests upon two additional samples shall be made and the average of the three tests shall determine the acceptance or rejection of the coil. For wire whose nominal diameter is between listed sizes, the requirements shall be those of the next larger size given in Table I.

TABLE I.—MINIMUM REQUIREMENTS FOR TENSILE STRENGTH AND ELONGATION.

Diameter, in.	Area		Tensile Strength, min., lb. per sq. in.								Elongation, min., per cent	
	Circular mils	Square Inch	Grades Nos. 9 and 13	Grades Nos. 15 and 20	Grade No. 30	Grade No. 40	Grade No. 55	Grade No. 65	Grade No. 80	Grade No. 85	in 10 in.	in 60 in.
0.2893	83 690	0.06573....	102 500	109 500	74 000	73 400	76 000	67 500	72 000	68 500	2.17	....
0.2576	66 370	0.05213....	108 800	114 500	80 000	74 900	77 800	68 500	73 500	69 900	1.98	....
0.2294	52 640	0.04134....	112 500	118 500	85 000	76 300	79 300	69 400	75 000	71 200	1.79	....
0.2043	41 740	0.03278....	115 200	121 300	89 000	77 700	80 900	70 200	76 400	72 500	....	1.24
0.1819	33 100	0.02600....	117 200	123 300	92 500	79 000	82 400	71 000	77 500	73 600	....	1.18
0.1620	26 250	0.02062....	119 000	125 000	94 800	80 400	84 000	72 000	78 600	74 700	....	1.14
0.1443	20 820	0.01635....	120 300	126 500	96 500	81 800	85 500	72 900	79 800	75 800	....	1.09
0.1285	16 510	0.01297....	121 600	127 900	97 900	83 200	87 000	74 200	81 000	77 000	....	1.06
0.1144	13 090	0.01028....	122 800	129 200	99 000	84 600	88 500	74 500	82 200	78 100	....	1.02
0.1019	10 380	0.008155...	123 900	130 300	100 100	86 000	90 000	75 100	83 400	79 200	....	1.00
0.09074	8 234	0.006467...	124 800	131 200	101 200	87 100	91 300	75 600	84 600	80 300	....	0.97
0.08081	6 530	0.005129...	125 600	132 000	102 200	88 200	92 600	76 200	85 700	81 400	....	0.95
0.07196	5 178	0.004067...	126 200	132 600	103 000	89 600	93 800	76 800	86 800	82 500	....	0.92
0.06408	4 107	0.003225...	126 700	133 200	103 700	90 400	95 000	77 400	87 800	83 400	....	0.90
0.05707	3 257	0.002558...	127 200	133 600	104 400	91 400	96 000	77 700	88 700	84 200	....	0.89
0.05082	2 583	0.002028...	127 500	134 000	105 200	92 100	97 000	78 100	89 500	85 000	....	0.87
0.04526	2 048	0.001609...	127 800	134 250	105 900	93 100	98 000	78 600	90 300	85 700	....	0.86
0.04030	1 624	0.001276...	128 000	134 500	106 600	94 000	98 600	79 000	91 000	86 400	....	0.85
0.03589	1 288	0.001012...	128 200	134 750	107 300	94 500	99 000	79 300	91 600	87 000	....	0.84
0.03196	1 022	0.0008023..	128 500	135 000	108 000	95 000	99 500	79 600	92 200	87 600	....	0.82

(*c*) Tests upon a section of wire containing a drawn joint shall show at least 95 per cent of the tensile strength of the unjointed wire. Elongation tests are not to be made upon test sections including joints.

Resistivity

8. Electric resistivity shall be determined upon fair samples by resistance measurements. The resistivity at a temperature of 20 C. (68 F.) shall not be more than the values shown for the designated grade, as follows:

GRADE	MAXIMUM RESISTIVITY, POUNDS PER MILE OHM	MAXIMUM RESISTIVITY, OHMS PER CIRCULAR MIL FOOT
No. 9..................................	9724	115.22
No. 13..................................	6732	79.78
No. 15..................................	5835	69.14
No. 20..................................	4376	51.86
No. 30..................................	2917	34.57
No. 40..................................	2188	25.93
No. 55..................................	1591	18.86
No. 65..................................	1346	15.96
No. 80..................................	1094	12.96
No. 85..................................	1030	12.20

Permissible Variations in Diameter

9. (*a*) The size shall be expressed as the diameter of the wire in decimal fractions of an inch, using not more than three places of decimals; that is, in mils.

(*b*) Wire is expected to be accurate in diameter; permissible variations from nominal diameter shall be as follows:

	OVER	UNDER
For wire 0.057 in. in diameter and larger...........	3 per cent	1 per cent
For wire less than 0.057 in. in diameter...........	1 mil	1 mil

(*c*) Each coil may be gaged at three places, one near each end and one approximately at the middle; the coil may be rejected if two points being within the accepted limits, the third point exceeds 4 per cent over, or 2 per cent under the nominal gage in the case of wire 0.057 in. in diameter or larger; or if the third point exceeds 1½ mils over or under the nominal gage in the case of wire less than 0.057 in. in diameter.

Density

10. For the purpose of calculating weights, cross-sections, etc., the density of the various alloys shall be taken as follows, based on a temperature of 20 C. (68 F.):

GRADE	DENSITY
Nos. 9 and 13..............................	8.78 g. per cu. cm., (0.3172 lb. per cu. in.)
No. 15[a]..................................	8.54 g. per cu. cm., (0.3085 lb. per cu. in.) 8.89 g. per cu. cm., (0.3212 lb. per cu. in.)
Nos. 20, 30, 40, 55, 65, 80 and 85............	8.89 g. per cu. cm., (0.3212 lb. per cu. in.)

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[a] The lower density value shall be used for alloys of the copper-tin-aluminum type. The higher density value shall be used for the copper-tin alloys.

Finish

11. The wire shall be free from all imperfections not consistent with the best commercial practice.

Packing and Shipping

12. (*a*) Package sizes for round wire shall be agreed upon in the placing of individual orders.

(*b*) The wire shall be protected against damage in ordinary handling and shipping.

Inspection

13. All testing and inspection shall be made at the place of manufacture. The manufacturer shall afford the inspector representing the purchaser all reasonable facilities to enable him to satisfy himself that the material conforms to the requirements of these specifications.

EXPLANATORY NOTE

NOTE.—It is the intention of these specifications to permit under each of the ten grades listed in Section 3 any commercial alloy coming within the total range of analysis given in Section 4, provided the product complies with the other requirements of these specifications. For purposes of information only, the types of alloy now commonly used for each of the several grades are listed below. The chemical composition of any of the materials shall be within the total range required in Section 4, but in no case will the alloy contain the allowed maximum of more than one constituent other than copper.

GRADE	ALLOY TYPE
No. 9	Copper, Silicon, Iron Copper, Silicon, Manganese Copper, Silicon, Zinc Copper, Silicon, Tin, Iron Copper, Silicon, Tin, Zinc
No. 13	Copper, Tin, Aluminum Copper, Tin, Silicon Copper, Tin, Silicon, Aluminum
No. 15	Copper, Silicon, Aluminum Copper, Tin Copper, Tin, Aluminum Copper, Tin, Aluminum, Silicon Copper, Tin, Silicon
No. 20	Copper, Tin
No. 30	Copper, Tin
No. 40	Copper, Tin Copper, Tin, Cadmium
No. 55	Copper, Tin, Cadmium
No. 65	Copper, Tin Copper, Tin, Cadmium
No. 80	Copper, Cadmium
No. 85	Copper, Cadmium

TENTATIVE SPECIFICATIONS

FOR

ROLLED ZINC[1]

A.S.T.M. Designation: B 69 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover three types of commercial rolled zinc, as described in Section 2. It should be understood that the specifications are general. Any closer limitations on permissible variations shall be a matter of agreement between the purchaser and the seller at the time of purchase.

SPECIFICATIONS

Types

2. The rolled zinc shall be divided into the following types:

Type I.—Coils or sheets cut from strip (ribbon) rolled zinc,
Type II.—Pack rolled zinc, and
Type III.—Zinc plates, such as boiler and hull plates produced by any rolling method.

Permissible Variations in Thickness

3. The permissible variations in thickness of rolled zinc shall be as follows:

	Thickness, in.	Permissible Variations in Thickness
Type I	Up to and including 0.020	±0.001 in.
	Over 0.020	±5 per cent
Type II	Up to and including 0.032	±0.002 in.
	Over 0.032	±6 per cent
Type III	All thicknesses	±6 per cent

Permissible Variations in Width

4. The permissible variations in width of all types of rolled zinc shall be as follows:

Thickness, in.	Permissible Variations in Width: Widths up to and Including 18 in.	Permissible Variations in Width: Widths over 18 in.
Up to 0.125	±0.031 in.	±0.063 in.
0.126 to 0.250	±0.063 in.	±0.063 in.
0.251 to 0.375	±0.125 in.	±0.125 in.
0.376 to 1.500	±0.250 in.	±0.250 in.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee B-2 on Non-Ferrous Metals and Alloys.
These Tentative Specifications are in effect a tentative revision of, and will supersede when adopted as standard, the present Standard Specifications for Rolled Zinc (A.S.T.M. Designation: B 69 – 29), 1936 Book of A.S.T.M. Standards, Part I, p. 705.

Permissible Variations in Length

5. The permissible variations in length of all types of rolled zinc shall be as follows:

(*a*) Sheets, strips and plates may be ordered to exact lengths up to 12 ft., in which case the following variations in length are permitted:

THICKNESS, IN.	PERMISSIBLE VARIATIONS IN LENGTH
Up to 0.500	±0.125 in.
0.500 to 1.500	±0.250 in.

(*b*) When plates, sheets, and strips are ordered in stock lengths, the pieces shipped shall be cut to the nominal or stock length as a maximum. The shortest acceptable lengths, the maximum permissible percentage by weight of short lengths, and the required percentage by weight of stock lengths in any one shipment shall be as shown in Table I.

TABLE I.—PERMISSIBLE VARIATIONS IN LENGTH OF PLATES, SHEETS, AND STRIPS ORDERED IN STOCK LENGTHS.

Nominal or Stock Length, ft.	Required Percentage by Weight of Stock Lengths	Maximum Permissible Percentage by Weight of Short Lengths				
		Over 8 to 10 ft.	Over 6 to 8 ft.	Over 4 to 6 ft.	2 to 4 ft.	Under 2 ft.
10	60	40	30	20	10	0
8	70	..	30	20	10	0
6	80	..	..	20	10	0

Coils

6. When Type I rolled zinc is supplied in coils it shall be coiled in a manner consistent with good commercial practice and in no case shall a coil consist of more than four pieces. No piece shall be less than 10 ft. in length.

Bend and Curvature

7. Type I rolled zinc in lengths over 10 ft. shall not exhibit sidewise bend or curvature in excess of 1 in. in any length of 10 ft.

Packing

8. (*a*) All material shall be properly separated according to size when loaded for shipment.

(*b*) Plates, sheets, and strips shall be packed in boxes, cases, or crates so constructed, and bulk shipments shall be so braced in the car, as to insure acceptance by common, or other, carriers for safe transportation at the lowest rate to the point of delivery.

Marking

9. Each bundle, box, or crate shall be legibly marked with dimensions and gross, tare, and net weights.

METHODS OF TESTING

Sidewise Bend or Curvature

10. The coil shall be supported on a reel at one end of a table and the ribbon drawn along a 10-ft. straight edge, keeping both ends of the straight edge touching the edge of the ribbon. No point on the straight edge shall then be more than 1 in. from the edge of the ribbon.

Gaging

11. Type I rolled zinc shall be gaged with a micrometer caliper which has previously been checked with precision blocks. No two measurements along the length of the coil shall be made within 12 in. of each other, nor shall measurements in any one line across the width of the coil be used as a basis of rejection.

APPENDIX

The following sections describing tests used to some extent in the rolled zinc trade, together with the tables which indicate roughly the range of properties obtainable as shown by the two test methods described, are included for information only:

DYNAMIC DUCTILITY TEST

A1. *Apparatus.*—The dynamic ductility machine for conducting the dynamic ductility test is a modified inclinable power press fitted with an adjustable die as shown in Fig. 1. The speed of the machine shall be 85 to 90 oscillations of the plunger per minute.

A2. *Test Specimens.*—(*a*) The test specimen shall consist of a strip cut across the full width of a ribbon or sheet, the width of the strip being approximately 3½ in. When this results in a specimen of less than 12 in. in length, as from narrow unslit ribbon zinc, two specimens 3½ in. in width shall be cut.

(*b*) Test specimens from narrow strips such as ribbon or sheet zinc after slitting shall be cut to 20-in. lengths.

NOTE.—The construction of the machine will not permit test specimens of greater thickness than 0.060 in.

A3. *Procedure.*—(*a*) The temperature of the test specimen, testing machine, and room throughout the test shall be kept within 70 and 90 F. The specimen shall be inserted in the machine and firmly clamped. The testing shall be started at one end and the strip moved with each successive cup. The series of cups shall be along the middle line of a 3½-in. strip so that clamping will be uniform around the cup as it is formed. With the motor running, the clutch shall be thrown in, lowering the plunger, which, with the die in the clamp base, presses the cup in the test strip. If the cup does not show rupture, the plunger shall be advanced by increments of 0.005 in. and the cupping repeated with each increased depth until rupture occurs. Having determined the rupturing point additional cups shall be formed as a check. The rupturing point shall not be considered to be that point where an open crack is formed, but where the drawing of the bottom of the cup has just caused "necking" of the surface prior to rupture (see Fig. 2).

NOTE.—An experienced operator should be able to determine the point of rupture after two or three trial cups.

(*b*) The depth of the cup at which rupture just occurs shall be read from the micrometer attachment on the plunger and reported as the dynamic ductility of the specimen. The average ductility of the test specimens shall be considered the dynamic ductility of the shipment.

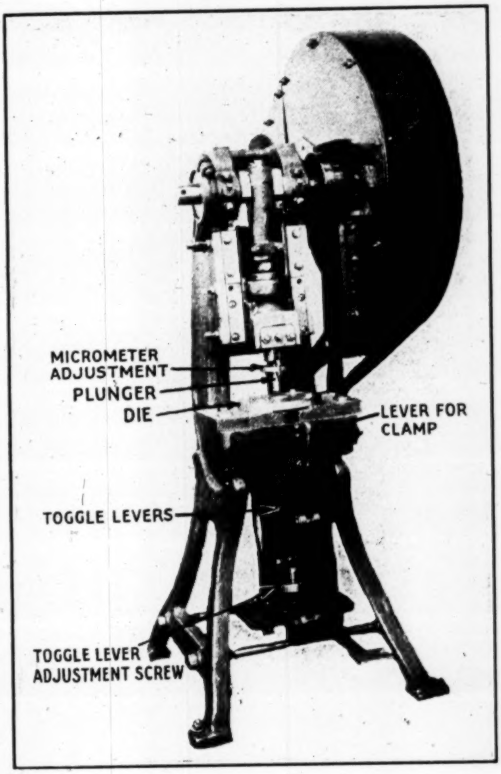


FIG. 1.—Dynamic Ductility Test Machine.

NOTE.—The micrometer attachment on the plunger mechanism measures the distance that the plunger extends beyond the zero point. The zero point is where no impression would be made on the test strip. The clamp base is adjusted for the varying thicknesses. The micrometer attachment also serves to advance and withdraw the plunger.

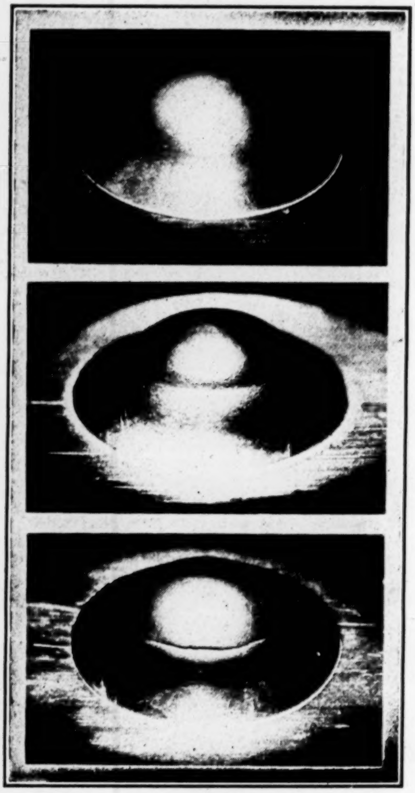
TEMPER TEST

A4. ***Apparatus.***—The apparatus consists of a temper test machine as shown in Fig. 3.

A5. *Test Specimens.*—Duplicate test specimens shall be cut from each sample and shall be rectangular in shape and cut accurately to 1$\frac{1}{2}$ in. in width and 4$\frac{31}{32}$ in. in length, with the length taken in the direction of rolling. With ribbon zinc the duplicate test specimens shall be cut from adjacent places across the width of a sample.

NOTE.—The construction of the machine does not permit test specimens of greater thickness than 0.035 in.

A6. *Procedure.*—(*a*) The temperature of the test specimens, testing machine, and room throughout the test shall be kept within 70 and 90 F. The specimens shall be inserted to the full depth of the vise of the mandrel and securely held by tightening the vise. The diameter of the mandrel is 2½ in. Inserting the test specimen as specified to the full depth of the vise leaves extended a length exactly one-half the circumference of the mandrel. In the rotation of the mandrel the test specimen is pressed by the contact arm against the mandrel. When the outer or free end passes the contact arm it springs out according to its temper. The speed of rotation of the mandrel shall be between 50 and 55 r.p.m. With the motor running, the clutch shall be thrown in, rotating the mandrel counterclockwise. When the outer end of the specimen has passed the contact arm the rotation of the mandrel shall be stopped by throwing out the clutch. The mandrel shall be turned to the 100 per cent position which is indicated by a mark on the mandrel and the fixed pointer, and the percentage of temper shall be read from the position of the outer end of the specimen on the underlying graduated scale, consisting of a half circle graduated counterclockwise from 0 to 100 per cent.



(*a*) Before Rupture.

(*b*) At Rupture.

(*c*) Past Rupture.

FIG. 2.—Showing Typical Cups Formed in Ductility Test.

TABLE AI.—DYNAMIC DUCTILITY AND TEMPER, TYPE I ROLLED ZINC.

American Zinc Gage	Thickness, in.	Dead Soft		Soft		Half Hard		Hard	
		Dynamic Ductility, min., in.	Temper, max., per cent	Dynamic Ductility, min., in.	Temper, max., per cent	Dynamic Ductility, min., in.	Temper, max., per cent	Dynamic Ductility, min., in.	Temper, max., per cent
No. 3	0.006	0.275	70	0.255	76	0.215	80	0.165	86
No. 4	0.008	0.280	65	0.260	72	0.220	75	0.170	83
No. 5	0.010	0.285	60	0.265	66	0.225	70	0.175	80
No. 6	0.012	0.290	55½	0.270	62	0.230	65	0.180	77
No. 7	0.014	0.295	52	0.275	58	0.235	61	0.185	74
No. 8	0.016	0.300	48½	0.280	54	0.240	58	0.190	72
No. 9	0.018	0.305	46	0.285	51	0.245	54	0.195	69
No. 10	0.020	0.310	43½	0.290	48	0.250	51	0.200	68
No. —	0.022	0.315	41½	0.295	45	0.255	48	0.205	65
No. 11	0.024	0.320	40	0.300	43	0.260	45	0.210	63
No. —	0.026	0.325	39	0.305	40	0.265	43½	0.215	61
No. 12	0.028	0.330	37½	0.310	37	0.270	42½	0.220	59
No. —	0.030	0.335	37	0.315	37	0.275	42	0.225	58
No. 13	0.032	0.340	36½	0.320	36½	0.280	41	0.230	57
No. —	0.034	0.345	36	0.324	36	0.285	40½	0.235	56
No. 14	0.036	0.347	..	0.326	..	0.290	..	0.240	..
No. —	0.038	0.349	..	0.328	..	0.295	..	0.245	..
No. 15	0.040	0.350	..	0.329	..	0.300	..	0.250	..
No. —	0.042	0.352	..	0.330	..	0.300	..	0.250	..
No. —	0.044	0.353	..	0.331	..	0.305	..	0.255	..
No. —	0.046	0.354	..	0.332	..	0.305	..	0.255	..
No. —	0.048	0.355	..	0.333	..	0.310	..	0.260	..
No. 17	0.050	0.356	..	0.334	..	0.310	..	0.260	..

TABLE AII.—DYNAMIC DUCTILITY AND TEMPER, TYPE II ROLLED ZINC.

		COMMERCIAL	
AMERICAN ZINC GAGE	THICKNESS, IN.	DYNAMIC DUCTILITY, MIN., IN.	TEMPER, MAX., PER CENT
No. 3	0.006	0.165	83
No. 4	0.008	0.170	77½
No. 5	0.010	0.175	73
No. 6	0.012	0.180	69
No. 7	0.014	0.185	65
No. 8	0.016	0.190	62
No. 9	0.018	0.195	58½
No. 10	0.020	0.200	57
No. —	0.022	0.205	55½
No. 11	0.024	0.210	54
No. —	0.026	0.215	52½
No. 12	0.028	0.220	51
No. —	0.030	0.225	49
No. 13	0.032	0.230	48
No. —	0.034	0.235	47
No. 14	0.036	0.240	..
No. —	0.038	0.245	..
No. 15	0.040	0.250	..
No. —	0.042	0.250	..
No. —	0.044	0.255	..
No. —	0.046	0.255	..
No. —	0.048	0.260	..
No. 17	0.050	0.260	..

(*b*) The duplicate specimen shall be similarly tested and, in order to eliminate the effect of the curvature of the specimens due to coiling, shall be inserted in the vise in the reverse position to that of the first specimen. That is, if one specimen is inserted and pressed with its curvature toward the mandrel, the duplicate specimen should be inserted with its curvature away from the mandrel.

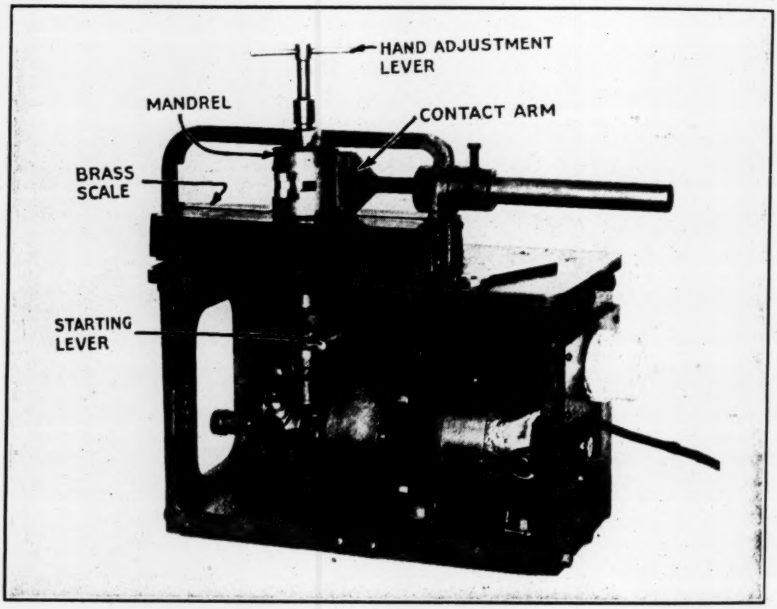


FIG. 3.—Temper Test Machine.

(*c*) The average of the readings on two test specimens shall be reported as the temper of the material.

DYNAMIC DUCTILITY AND TEMPER OF COMMERCIAL ZINC

A7. Tables AI and AII are illustrative of the properties which may be found in commercial grades of rolled zinc of Types I and II, respectively.

TENTATIVE SPECIFICATIONS
FOR
ALUMINUM-BASE ALLOY DIE CASTINGS[1]

A.S.T.M. Designation: B 85 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1931; REVISED, 1933, 1937.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

Scope

1. These specifications cover die castings die-cast from aluminum-base alloys. Eight alloy compositions are specified, designated alloys Nos. IV to IX, inclusive, and Nos. XI and XII.

Chemical Composition

2. The alloys shall conform to the requirements as to chemical composition given in Table I within the limits specified therein.

3. (*a*) It is understood that iron contributes to the hardness in all these alloys. While the use of steel or iron containers for the commercial manufacture of die castings unavoidably introduces some of this element into the alloy, it shall not exceed the percentages specified in Table I.

(*b*) Manganese and nickel are not regarded as harmful elements in these alloys up to the limits specified in Table I.

Sampling

4. Samples for chemical analysis may be taken either by sawing, drilling, or milling a representative group of castings, and shall represent the average cross-section of the material. The castings and cutter shall be thoroughly cleaned and no lubricant shall be used during the operations.

Chemical Analysis

5. Chemical analysis shall be made in accordance with the

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee B-6 on Die-Cast Metals and Alloys.

Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys (A.S.T.M. Designation: B 40 – 36 T) of the American Society for Testing Materials.[1]

Density

6. No casting shall be acceptable which weighs less than a minimum agreed upon by the manufacturer and purchaser for the individual order.

Workmanship and Finish

7. Castings shall be produced with smooth surfaces, free from cracks and disfiguring blemishes.

TABLE I.—CHEMICAL COMPOSITIONS OF ALUMINUM-BASE DIE-CASTING ALLOYS.

Alloy[a]	Desired Composition, per cent				Permissible Variations in Alloying Elements, per cent					Other Elements, max., per cent					
	Copper	Silicon	Nickel	Aluminum	Copper	Silicon	Copper and Silicon, max.	Nickel	Copper, Silicon and Nickel, max.	Iron	Zinc	Manganese	Magnesium	Tin	Total, Other Impurities[b]
No. IV..	...	5	...	95	0.6 max.	4.5 to 6.0	...	0.5 max.	...	2.0	0.5	0.3	0.10	0.1	0.2
No. V...	...	12	...	88	0.6 max.	11.0 to 13.0	...	0.5 max.	...	2.0	0.5	0.3	0.10	0.1	0.2
No. VI..	2	3	...	95	1.5 to 2.5	2.5 to 3.5	...	0.5 max.	...	2.5	0.8	0.3	0.10	0.1	0.2
No. VII.	4	5	...	91	3.5 to 4.5	4.5 to 5.5	...	0.5 max.	...	2.3	0.8	0.3	0.10	0.1	0.2
No. VIII	1.5	1	2.25	95.25	1.0 to 2.0	0.8 to 2.0	...	1.8 to 2.5	...	2.5	0.8	0.3	0.10	0.1	0.2
No. IX..	4	1.75	4	90.25	3.5 to 4.5	1.0 to 2.5	...	3.5 to 4.5	11.0	1.8	0.8	0.3	0.05	0.1	0.2
No. XI..	2	8	...	90	1.0 to 3.0	7.0 to 9.0	11.0	0.5 max.	...	2.0	0.5	0.3	0.10	0.1	0.2
No. XII.	7	2	...	91	6.0 to 8.0	3.5 max.	11.0	0.5 max.	...	2.3	1.5	0.3	0.10	0.1	0.3

[a] The alloy designations correspond to numbers used in the investigation on aluminum-base die-casting alloys carried out under the jurisdiction of Committee B-6 on Die-Cast Metals and Alloys (formerly Subcommittee XV on Die-Cast Metals and Alloys of Committee B-2 on Non-Ferrous Metals and Alloys), the alloy numbers omitted correspond to alloy compositions which were investigated by the Committee, but which were not considered desirable of inclusion in these specifications. See *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part I, p. 192 (1929).

[b] Analysis shall ordinarily be made only for the elements specifically mentioned in this table. If, however, the presence of other elements is indicated in the course of routine analysis, further analysis shall be made to determine that these other elements do not exceed the total given in the last column.

Permissible Variations in Dimensions

8. Dimensional tolerances shall be within the limits specified on the drawings describing the casting or castings on order, or shall be within the limits specified in the order.

Rejection

9. Castings which show injurious defects subsequent to acceptance may be rejected. The manufacturer's liability shall be confined to replacing rejected castings without charge to the purchaser.

Physical Properties and Tests.

10. Unless specified in the order or specifically guaranteed by the manufacturer, acceptance of castings under these specifications shall not depend on physical properties determined by tension or impact tests.

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[1] **A.S.T.M. Methods of Chemical Analyses of Metals, p. 95 (1936).**

APPENDIX

The data in the following table do not constitute a part of these specifications. They will indicate to the purchaser the physical properties that may be expected of tension and impact test specimens when made in a die, the test specimens being cast in regular production routine and conforming to the chemical composition specified. It should be thoroughly understood that the following figures represent die-cast test specimens and not specimens cut from commercial die-cast parts. Rockwell hardness values have not been shown because they are considered too unreliable on die castings.

Alloy	Tension Tests on Round Specimens		Charpy Impact on Square Specimens, ft-lb.
	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	
No. IV	29 000	3.5	4.5
No. V	33 000	1.5	2.0
No. VI	30 000	3.5	5.0
No. VII	32 000	2.0	2.5
No. VIII	29 000	4.0	4.5
No. IX	31 000	1.5	2.0
No. XI	32 000	1.7	3.0
No. XII	34 000	1.6	3.2

TENTATIVE SPECIFICATIONS
FOR
MAGNESIUM-BASE ALLOY DIE CASTINGS[1]

A.S.T.M. Designation: B 94 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1934; REVISED, 1937.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

Scope

1. These specifications cover magnesium-base alloy die castings having a specific gravity of about 1.8. One alloy is specified and is designated as Alloy No. 12.

Manufacture

2. The alloy may be made from virgin metal or from purified scrap metal of known composition.

Chemical Composition

3. The castings shall conform to the following requirements as to chemical composition:

Magnesium, min., per cent		remainder
Aluminum, per cent		9.0 to 11.0
Silicon, max., per cent		1.0
Manganese, min., per cent		0.10
Copper, max., per cent		0.05
Nickel, max., per cent		0.03
Other ingredients (zinc, cadmium, tin)	Individually, max., per cent	0.3
	Total, max., per cent	0.5

Sampling

4. (*a*) Samples for chemical analyses shall be taken either by sawing, drilling, or milling a representative group of castings, and shall represent the average cross section of the material. The sample shall weigh not less than 50 g.

(*b*) The saw, drill, cutter, or other tool used shall be thoroughly cleaned. No lubricant shall be used in the sampling operation.

Workmanship and Finish

5. The castings shall be of uniform quality and shall be produced with smooth surfaces, free from cracks and disfiguring blemishes.

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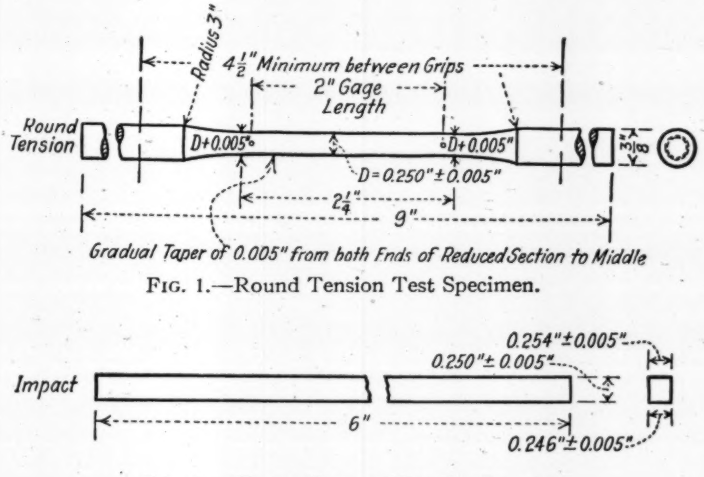
[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee B-6 on Die-Cast Metals and Alloys.

Rejection

6. Die castings which show injurious defects revealed by machining operations subsequent to acceptance may be rejected, and if rejected the manufacturer's responsibility shall be limited to replacing rejected castings without charge to the purchaser.

EXPLANATORY NOTES

NOTE 1.—The data in the following table covering Alloy No. 12 do not constitute a part of these specifications. They will indicate to the purchaser the physical properties that may be expected of tension and impact test specimens when made in a die, the test specimens being cast in regular production routine and conforming to the dimensions shown in Figs. 1 and 2 and to the chemical composition specified. Rockwell hardness values have not been shown because they are considered too unreliable on die castings.



Gradual Taper of 0.005" from both Ends of Reduced Section to Middle

FIG. 1.—Round Tension Test Specimen.

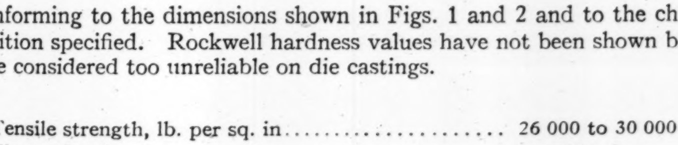


FIG. 2.—Charpy Impact Test Specimen.

Tensile strength, lb. per sq. in.	26 000 to 30 000
Elongation in 2 in., per cent	1 to 3
Charpy impact, ft-lb	1 to 3

NOTE 2.—Alloy No. 12, as specified in these specifications, corresponds to Alloy No. 2 in the Tentative Specifications for Magnesium-Base Alloy Ingot for Remelting (B 93 – 36 T) of the American Society for Testing Materials.[1]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 706 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 340.

The aluminum content has been set at 9.0 to 11.0 per cent. Alloys with lower aluminum content have somewhat higher percentage elongation and Charpy impact values but have lower fluidity and, hence, are more difficult to cast. Alloys with higher aluminum content are too brittle for many purposes.

Manganese is added to this type of alloy in order to improve corrosion resistance, particularly against salt water and salt-water atmosphere. Manganese is only slightly soluble in an alloy of this type and 0.10 per cent has been found sufficient to obtain the desired results. The alloy will usually contain 0.15 to 0.30 per cent manganese, although a manganese content in excess of this amount is not detrimental.

The copper and nickel contents are set at 0.05 and 0.03 per cent, maximum, respectively, inasmuch as larger percentages of these elements decrease the corrosion resistance of the alloy.

The present phraseology for expressing the composition of these magnesium-base die castings permits the allowable presence of appreciable amounts of metals such as zinc, cadmium, and tin that are often present in other magnesium-base alloys and which, in small quantities, have no detrimental effects on magnesium-base alloy die castings. Such metals, therefore, should not be classed as impurities.

TENTATIVE METHOD OF TEST
FOR
FLEXIVITY OF THERMOFLEX (THERMOSTATIC METALS)[1]

A.S.T.M. Designation: B 106 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1936; REVISED, 1937.

Scope

1. (*a*) This method is intended to cover the determination of the flexure-temperature characteristics of thermoflex (thermostatic metals) in the form of flat strips.

(*b*) The characteristics of thermoflex are determined as a whole when tested in the form of a flat strip. No account is taken of the characteristics of the individual components.

(*c*) The procedure involves changing the temperature of a strip of thermoflex and obtaining values indicating the response to temperature changes, or the flexivity, by calculation from the temperatures, dimensions of specimen, and relative movement of gage points on the specimen.

Definitions

2. The following definitions and symbols shall apply in connection with the tests as outlined in this method:

(*a*) *Thermoflex.*—Thermoflex is a composite material usually in the form of sheet or strip, comprising two or more materials of any appropriate nature, metallic or otherwise, which, by virtue of the differing expansivities of its components, tends to change its curvature when subjected to a uniformly distributed change of temperature.

(*b*) *Component.*—A component is one of the plurality of more or less distinct laminae that compose a given thermoflex, and which in cooperation with the other components has a determining effect on the sensitivity to temperature of the thermoflex in so far as tendencies to curvature change are concerned.

(*c*) *Deflection* (*c*).—Deflection as applied to thermoflex is the movement due to temperature change of a chosen point or line thereon, relative to other points or lines, regarded as fixed.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys.

(*d*) *Deflection, D.*—For the purposes of this test the deflection, D, of a thermoflex strip is the perpendicular distance from a point on the longitudinal center line of the specimen to a line defined by the points of intersection of the longitudinal center line of the specimen with planes passing through the knife edges perpendicularly to the plane of the latter; the first mentioned point being equidistant from the other two.

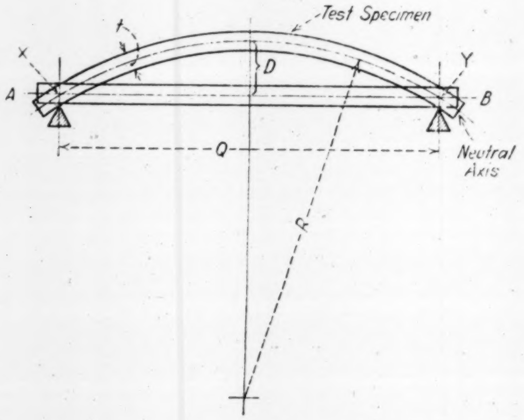

FIG. 1.—Test for Flexivity of Thermoflex.

(*e*) *Flexivity, F.*—The flexivity, F, is the change of curvature of the longitudinal center line of the specimen per unit temperature change for unit thickness, and is given by the formula:

$$F = \frac{\left(\frac{1}{R_1} - \frac{1}{R_0}\right)t}{T_1 - T_0} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

whence, referring to Fig. 1:

$$F = \frac{8t}{T_1 - T_0}\left(\frac{D_1}{Q^2 + 4D_1t + 4D_1^2} - \frac{D_0}{Q^2 + 4D_0t + 4D_0^2}\right) \ldots\ldots\ldots (2)$$

where R = the radius of curvature, in inches,
t = the thickness, in inches,
D = the deflection, in inches, as measured with reference to the plane including the line AB and parallel to the knife edges (see Fig. 1).
Q = the distance between knife edges, in inches,
T = the temperature, in degrees Fahrenheit,
$T_1 - T_0$ = the change in temperature in degrees Fahrenheit,
D_0 = D corresponding to T_0, and
D_1 = D corresponding to T_1.

Equation (2) is an exact expression which takes into account initial curvature. If the specimen is accurately straight at T_0, then $\frac{1}{R_0}$ in Eq. (1) is zero, D_0 is zero, and Eq. (2) reduces to:

$$F = \frac{8t}{T_1 - T_0}\left(\frac{D_1}{Q^2 + 4D_1 t + 4D_1^2}\right) \qquad \text{(3)}$$

Test Specimen

3. (*a*) The test specimen shall be in the form of a strip which displays no apparent initial irregularity of curvature.

(*b*) The maximum thickness shall not be greater than the minimum thickness by more than 1 per cent of the latter.

(*c*) The width shall not be less than five times nor more than ten times the thickness. The maximum width shall not be greater than the minimum width by more than 2 per cent of the latter.

(*d*) The length shall be such as to allow a distance between knife edges not less than ten times nor more than fifteen times the mean width, and to extend beyond the knife edges a distance not less than the width. The ratio of the distance between knife edges to the thickness shall be determined to an accuracy of 0.10 per cent.

(*e*) Dimensions of specimens shall be determined by means of screw micrometers or by equivalent methods, to an accuracy of 1 per cent.

NOTE.—For specimens less than 0.050 in. in thickness special precautions are necessary, such as the use of a micrometer reading directly to 0.0001 in., or suitable optical methods may be used.

In the case of very thin specimens (less than 0.010 in. in thickness) the average thickness may be calculated from measurements of length, average width, weight and density. When the density is unknown, it may be determined by weighing a sample of at least 10 g., first in air and then in water. The density in grams per cubic centimeter is equivalent to the weight in air divided by the loss of weight due to submergence in water. The temperature of the water shall be approximately the same as that of the balance room to avoid errors due to convection currents. For the accuracy required, no correction for the temperature of the water or the buoyancy of the air is required. However, care shall be exercised to remove all air bubbles from the sample when weighing it in water, and to avoid the presence of grease or other films on the surface of the water. To this end it is recommended that after a preliminary cleaning to remove obvious dirt the sample be attached to a fine wire, to be used later in suspending it while weighing, and thoroughly rinsed, first in ether, then alcohol, and finally water, before immersing in the water to be used for weighing.

Apparatus

4. The apparatus shall consist of the following:

(*a*) *Specimen Table.*—A specimen table carrying two parallel knife edges to support the specimen. The distance between the knife edges shall be known to within 0.2 per cent.

(*b*) *Micrometer.*—A micrometer, traveling microscope or equivalent device so connected to the specimen table that expansion during heating of the table or connecting parts will not cause appreciable displacement of the measuring device with respect to the knife edges.

(*c*) *Bath.*—A stirred liquid bath or uniformly heated enclosure, in which the specimen table together with adjustable electric heating source is placed, by means of which the specimen can be maintained at the desired temperatures, with a variation in temperature throughout the gage length of the specimen of not to exceed 0.5 per cent of the temperature range used in the test.

(*d*) *Thermocouple or Thermometer.*—Thermocouple wire of known calibration, or calibrated mercury-in-glass thermometers for measuring the temperature of the specimen.

(*e*) *Potentiometer.*—Means for determining the electromotive force of the thermocouples with an accuracy of 1 per cent, and of such sensitivity as to enable the detection of temperature changes of $\frac{1}{4}$ per cent of the temperature range involved in the test.

(*f*) *Transmission Rod.*—Means for measuring the deflection of the strip at a point midway between the knife edges. For this purpose there may be used a fused quartz "transmission rod" terminating in a point or knife edge to engage the specimen at a point midway between the points of contact with the knife edges. This rod shall be suspended from the specimen or otherwise supported with its axis normal to the plane of the knife edges in such a manner that it remains free to move in the direction of its axis. This rod shall bear at its free end an index suitable for microscopic observation, or an electrical contact to permit the changes in the deflection of the specimen to be accurately measured. Alternatively, the deflection of the specimen may be directly observed by optical means whose line of sight is in the plane equidistant from the knife edges and perpendicular to the plane of the latter.

(*g*) The transmission rod and any attached parts shall be of such weight or so counterweighted that they will not cause a deflection greater than 1 per cent of the maximum to be produced by the action of the thermoflex alone. When free, the thermoflex assumes very nearly a circular curvature. Concentrated loading at the center of the specimen will cause the curvature to be other than circular, and may cause significant errors in the evaluation of flexivity. The location of the plane of the knife edges with reference to the scale of the micrometer shall be known to within 0.5 per cent of the maximum deflection.

Preparation of Specimen

5. (*a*) After being roughly cut or sheared from the sample, the specimen shall be finished to size by careful machining or filing. An amount of material extending a distance not less than three times the thickness shall be removed along each edge of the specimen, to eliminate material which may have been damaged in cutting.

(*b*) When the specimen has been finished to size, any necessary reference marks shall be made (by such means as a sharp drill, scribing tool or milling cutter) and their relative locations determined and recorded. Center punches or similar means shall not be used because of the distortion produced.

Procedure

6. (*a*) *Stabilization.*—After all preparatory work on the specimen has been completed, it shall be subjected to a stabilizing heat treatment to relieve internal stresses. This treatment may consist in heating the specimen, while free to bend, for a prescribed time and temperature. The details of the stabilizing procedure will depend upon the characteristics of the thermoflex being tested and shall be as mutually agreed upon by the manufacturer and purchaser.

(*b*) *Test Routine.*—The specimen shall be mounted on the knife edges of the specimen table, the transmission rod put in place and a zero reading taken at room temperature. Slight mechanical pressure shall be applied and then removed at a point near the center of the specimen. If appreciable zero shift is apparent with repeated applications of pressure, the cause shall be determined and removed before proceeding with the test.

(*c*) When satisfactory initial conditions have been established, observations of deflection and temperature shall be made at room temperature, and the results recorded.

(*d*) The temperature of the specimen shall then be adjusted to the next value desired. Thermocouple or thermometer observations at points on or near the center and ends of the specimen shall then be made and recorded.

(*e*) The deflection shall then be observed and recorded.

(*f*) Temperature observations as described in Paragraph (*d*) shall again be made and recorded. If significant discrepancies of temperature or its distribution are found, they shall be corrected and another deflection observation made and recorded.

(*g*) After having secured satisfactory temperature conditions and corresponding deflection data, the next chosen temperature shall be established and the above routine again followed and so on over the range of temperature agreed upon.

(*h*) In all cases, a final set of observations shall be made at or near room temperature to determine whether or not there has been permanent distortion or any mechanical incident that would prevent determination of deflectivity within the desired limits of accuracy. If such is evident, the test shall be repeated under one of the following conditions as agreed upon:

(*1*) The same specimen shall be tested over the same temperature range.

(*2*) The same specimen shall be tested over a different temperature range.

(*3*) Another specimen shall be tested over the same temperature range.

(*4*) Another specimen shall be tested over a different temperature range.

(*i*) *Record.*—Measurements and observations shall be recorded on a data sheet similar to that shown in Table I.

Report

7. The report shall include the following:

(*a*) Class of thermoflex,

(*b*) Identification of specimen,

(*c*) Dimensions of specimen,

TABLE I.—DATA SHEET FOR FLEXIVITY TEST OF THERMOFLEX.

Date.. Class of Thermoflex..

Laboratory Name.. Sample No..

Dimensions of Specimen:

Length..

Thickness, Average....................

Width.. Knife Edge Spacing..

Temperature Range:

.... F. to .... F. to .... F.

Time, sec.	Elapsed Time, sec.	Temperature of Specimens, deg. Fahr.	Deflection, in.	Remarks

First Cycle	Second Cycle
.... F. to .... F. to .... F.	.... F. to .... F. to .... F.

Permanent Set..

(*d*) Spacing of knife edges,

(*e*) Flexivity,

(*f*) A curve showing the relation between flexivity and temperature for each established temperature, and

(*g*) Amount of permanent set at the end of each temperature cycle with reference to original zero reading or room temperature.

TENTATIVE METHOD OF CHEMICAL ANALYSIS OF PORTLAND CEMENT[1]

A.S.T.M Designation: C 114 - 37 T

This is a Tentative Standard and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1934; Revised, 1935, 1937.[2]

Magnesia

Method A. Acid-Alkali Method for Rapid Determination of Magnesia[3]

Solutions Required

1. The following solutions will be required:

(*a*) *Trinitrobenzene Indicator.*—Dissolve 1.0 g. of c.p. 1-3-5 trinitrobenzene in 100 ml. of ethyl alcohol (95 to 100 per cent).

(*b*) *Phenolphthalein Indicator.*—Dissolve 0.4 g. of c.p. phenolphthalein in 100 ml. of ethyl alcohol (50 per cent).

(*c*) *Standard Sodium Hydroxide Solution.*—Dissolve 13.0 g. of sodium hydroxide in water, add 10 ml. of a freshly filtered saturated barium hydroxide solution, and dilute to 1 liter with distilled water which has been recently boiled and cooled. Shake the solution from time to time for several hours, and filter into a tubulature bottle fitted with an automatic burette. Protect the solution from carbon dioxide in the air, with soda-lime tubes at the top of the bottle and the burette.

The solution as prepared is approximately $\frac{1}{3}$ N, and shall be standardized against acid potassium phthalate or benzoic acid acidimetric standards furnished by the National Bureau of Standards (Standard Samples Nos. 84 and 39), using the methods in the certificates accompanying the standard samples. The exact normality of the solution shall be determined, and its MgO equivalent calculated on the basis that 1 ml. of a normal solution is equivalent to 0.02016 g. of MgO.

(*d*) *Standard Acid Solution.*—Dilute 32.0 ml. of HCl (sp. gr. 1.19) or 26.0 ml. of HNO_3 (sp. gr. 1.42) to 1 liter with distilled water. This solution will be slightly in excess of $\frac{1}{3}$ N, and shall be standardized against the standard sodium hydroxide solution, using phenolphthalein indicator, and adjusted by dilution until the two solutions are of equal strength.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] This method when adopted as standard is intended to be included with the procedures for chemical analysis appearing in Sections 8 to 17 of the Standard Methods of Sampling and Testing Portland Cement (A.S.T.M. Designation: C 77 - 37), 1937 Supplement to Book of A.S.T.M. Standards, p. 54.

[3] This procedure is a modification of the method originally published by Fred P. Diener in *Concrete*, Vol. 39, No. 6, December, 1931. The changes, which are in minor details and do not affect the underlying principles of the method, were made by the author in cooperation with the Working Committee on Methods of Chemical Analysis of Cement of the A.S.T.M. Committee C-1 on Cement for use in the investigations sponsored by the working committee. The theory of the method is discussed in the original article.

(*e*) *Potassium Iodide—Starch Paper.*—Mix equal volumes of a 5 per cent potassium iodide solution and a 1 per cent soluble starch solution, and dip strips of filter paper into the mixture.

Method

2. (*a*) To 5.0 g. of cement in a 600-ml. beaker shall be added about 50 ml. of water and 50 ml. of HCl (1:1), and the solution boiled until the cement is completely in solution, excepting possibly some silica which may separate out (Note 1). Then 5 ml. of saturated bromine water shall be added and the boiling continued until the excess bromine is completely expelled, as indicated by the absence of a blue coloration on potassium iodide - starch paper when held in the issuing steam. The solution shall then be diluted to about 250 ml. with hot water and a 20 per cent solution of sodium hydroxide added to the boiling solution from a pipette, until the excess acid is neutralized and a permanent precipitate just forms. Then, continuing the boiling, HCl (1:1) shall be carefully added from a pipette until the precipitate dissolves and the solution assumes a yellow ferric chloride color, avoiding much excess acid. The sides of the beaker shall be washed down with hot water and the boiling continued for a minute or two, and then the beaker shall be removed from the burner.

(*b*) To the solution shall be cautiously added 4.0 g. of calcium carbonate (weighed to the nearest 0.1 g.). This amount should be sufficient to completely neutralize the acid, with some excess. The solution shall then be diluted to about 400 ml. with hot water and boiled vigorously for 10 min. The solution shall be removed from the burner, and a 0.5 per cent potassium permanganate solution carefully added to the hot solution from a burette, with vigorous stirring, until the supernatant liquid shows a faint pink color after settling for a moment. Avoid use of more permanganate than necessary to produce a faint pink color. Then the boiling shall be continued for 2 min., 2.0 ml. of ethyl alcohol added, and the boiling continued for a few minutes or until the pink color has disappeared (Note 2).

(*c*) The contents of the beaker shall then be transferred to a 500-ml. volumetric flask, cooled to room temperature, diluted to the mark, and mixed by shaking. The solution shall be filtered through a dry paper, and after discarding the first 10 or 20 ml. of filtrate, 200 ml. of the filtrate shall be transferred with a pipette to a 500-ml. Erlenmeyer flask. The solution in the flask shall be heated to boiling, and 3.0 ml. of trinitrobenzene indicator added. Then standard sodium hydroxide solution shall be added 1.0 ml. at a time with intermittent boiling for a few seconds, until the magnesium is completely precipitated and a definite pink color persists on boiling. The total amount of alkali used shall be recorded (Note 3).

(*d*) The solution shall be transferred to a 250-ml. volumetric flask, cooled to room temperature, diluted to the mark, and mixed by shaking. The solution shall be filtered through a dry paper, into a graduated cylinder, discarding the first 10 or 20 ml. of filtrate, until 125 ml. of filtrate are secured. The 125 ml. of filtrate shall be transferred to a flask, a few drops of phenolphthalein indicator added, and the solution titrated with standard acid solution to the disappearance of the pink color.

(*e*) *Calculation.*—If the acid and alkali solutions are of equal strength, the quantity of alkali in milliliters minus twice the quantity of acid in milliliters is equivalent to the MgO in a 2-g. sample:

$$\text{MgO, per cent} = \frac{100\,(a - 2b)}{2} \times \text{MgO equivalent of 1 ml. standard alkali}$$

where a = the quantity of standard alkali in milliliters;

b = the quantity of standard acid in milliliters.

(*f*) *Blank Determination.*—A blank determination shall be made, using the same procedure as outlined above, and using approximately the same amounts of reagents. The titration results shall be corrected accordingly.

NOTE 1.—Some samples of cement, particularly those containing considerable iron or manganese, may require longer boiling or the addition of slightly more HCl to effect complete solution.

NOTE 2.—The purpose of the addition of potassium permanganate is to remove manganese from the sample, which otherwise would be precipitated and reported as MgO. The permanganate in a hot solution oxidizes the manganese to insoluble MnO_2 and is itself reduced to MnO_2. The ethyl alcohol precipitates the excess permanganate. In samples containing little or no manganese, this portion of the procedure may be omitted.

NOTE 3.—Enough standard alkali should be added to completely precipitate the magnesium without too large an excess which might cause some precipitation of calcium. A guide to proper procedure is the amount of back titration with acid, which should be held between 1.0 and 2.0 ml. (after being doubled). A little experience with the color change of the trinitrobenzene indicator will enable the operator to hold the back titration between these limits.

Method B. Rapid Determination of Magnesia by Titration of Magnesium Oxyquinolate[1]

Solutions Required

3. The following solutions will be required:

(*a*) *8-Hydroxyquinoline* (*1.25 per cent*).—Dissolve 25 g. of 8-hydroxyquinoline in 60 ml. of glacial acetic acid. When the solution is complete dilute to 2 liters with cold water. One milliliter of this solution is equivalent to 0.0016 g. of MgO.

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[1] This procedure is a modification of the procedure originally published by J. C. Redmond in the Bureau of Standards *Journal of Research*, Vol. 10, No. 6, June, 1933. The revision was made after correspondence with Mr. Redmond and for use in investigations sponsored by the Working Committee on Methods of Chemical Analysis of Cement of the A.S.T.M. Committee C-1 on Cement. The revised procedure differs from the original one mainly in two respects: (*1*) The hydrates of iron and aluminum are precipitated twice instead of once; and (*2*) the amounts of 8-hydroxyquinoline reagent and standard bromate solution used are variable instead of fixed.

The original article should be consulted for a discussion of the theory and experimental basis of the method. The method was developed primarily for use by the National Bureau of Standards for the detection of cements whose magnesia content did not meet the requirements of Federal Specifications Board Specification No. 1a. For use in the investigations of the working committee, the method was so revised that the magnesia content of any cement might be determined rapidly and correctly.

(*b*) *Sodium Thiosulfate Solution* (*0.1 N*).—Dissolve 25 g. of $Na_2S_2O_3 \cdot 5H_2O$ in 200 ml. of water and dilute to 1 liter. Standardize this solution either on sodium oxalate through a permanganate solution, or on arsenious oxide through a solution of resublimed iodine. Standard Samples Nos. 40 and 83 of the National Bureau of Standards are convenient for this purpose. One milliliter of exactly 0.1 *N* sodium thiosulfate solution is equivalent to 0.000504 g. of MgO.

(*c*) *Potassium Bromate - Potassium Bromide Solution* (*0.2 N*).—Dissolve 20 g. of KBr and 5.57 g. of $KBrO_3$ in 200 ml. of water and dilute to 1 liter. Obtain the ratio of this solution to the standard sodium thiosulfate solution as follows: To 200 ml. of water in a 400-ml. beaker add exactly 25 ml. of bromate solution. Add 20 ml. of HCl (sp. gr. 1.19), stir, and add immediately 10 ml. of KI solution. Mix well and titrate at once with the sodium thiosulfate solution until nearly colorless. Add 2 ml. of starch solution, and titrate to the disappearance of the blue color.

(*d*) *Potassium Iodide Solution* (*25 per cent*).—Dissolve 25 g. of KI in 100 ml. of water.

(*e*) *Starch Solution.*—To 500 ml. of boiling water add a cold suspension of 5 g. of soluble starch in 25 ml. of water, cool, add a cool solution of 5 g. of NaOH in 50 ml. of water, add 15 g. of KI and mix thoroughly.

Method

4. (*a*) To a 0.500-g. sample of cement in a 400-ml. beaker shall be added 10 ml. of water and 10 ml. of HCl (sp. gr. 1.19). This shall be heated gently and any coarse particles ground with the flattened end of a glass rod until decomposition is complete. Hot water shall then be added to make a total volume of 100 ml.

(*b*) Three drops of methyl red (0.02 alcoholic solution) shall be added to the solution and then NH_4OH(sp. gr. 0.90) added until the solution is distinctly yellow. This solution shall then be heated to boiling and boiled for 1 to 2 min., then removed from the hot plate and allowed to stand until the precipitate has settled. The solution shall then be filtered without delay and the precipitate washed twice with a hot solution (2 per cent) of ammonium chloride. The precipitate shall then be transferred with the filter paper to the beaker and dissolved in 10 ml. of HCl (1:1). This shall be diluted to about 100 ml. and heated to boiling. The hydrates shall then be reprecipitated, filtered, and washed as above. To the filtrate shall be added 1 ml. of NH_4OH (sp. gr. 0.90). The filtrate shall then be heated to boiling and while boiling 25 ml. of a hot solution (4 per cent) of $(NH_4)_2 C_2O_4 \cdot H_2O$ shall be added and the boiling continued for 2 min. The digestion shall be continued on the steam bath for 10 to 15 min.

(*c*) The solution shall be cooled to 70 C. and 10 to 25 ml. of the 8-hydroxyquinoline reagent added (Note 1) and then 4 ml. of NH_4OH

(sp. gr. 0.90) added per 100 ml. of solution. The solution shall be stirred on a mechanical stirring machine for 15 min. and set aside until the precipitate has settled (Note 2). The solution shall then be filtered and the precipitate washed with hot dilute NH_4OH (1:40). The precipitate shall then be dissolved in 50 to 75 ml. of the hot dilute HCl (1:9), the resulting solution diluted to 200 ml. and then 15 ml. of HCl (sp. gr. 1.19) added. The solution shall then be cooled to 25 C. and 10 to 35 ml. of standard bromate solution (Note 3) added from a pipette. The solution shall then be stirred and allowed to stand for about 30 sec. to insure complete bromination. To this solution shall then be added 10 ml. of the KI solution. The resulting solution shall be stirred well and then titrated with the standard sodium thiosulfate solution until the color of the iodine becomes faintly yellow. At this point 2 ml. of the starch solution shall be added and the solution titrated to the disappearance of the blue color.

(*d*) *Calculation.*—The percentage of magnesia in a 0.5-g. sample may be calculated as follows:

$$\text{Percentage of MgO} = [(B - C) \times A] \times 200$$

where A = the weight of MgO in grams per milliliter of standard sodium thiosulfate solution;

B = milliliters of standard sodium thiosulfate solution equivalent to 25 ml. of standard bromate solution, and

C = milliliters of sodium thiosulfate solution required in the titration.

(*e*) *Blank Determination.*—A blank determination shall be made, using the same procedure as outlined above, and using approximately the same amounts of reagents. The titration results shall be corrected accordingly.

NOTE 1.—An excess of the 8-hydroxyquinoline reagent should be used. If it is small the result of the determination of magnesia may be low; on the other hand, if large, the result may be high. The following guide may be used:

APPROXIMATE CONTENT OF MAGNESIA, PER CENT	APPROXIMATE AMOUNT OF REAGENT REQUIRED, ML.
0 to 1 5	10
1.5 to 3.0	15
3.0 to 4.5	20
4.5 to 6.0	25

NOTE 2.—The precipitate should be filtered within an hour. Prolonged standing may cause high result.

NOTE 3.—The amount of the standard bromate solution used may be as follows:

APPROXIMATE CONTENT OF MAGNESIA, PER CENT	AMOUNT OF STANDARD BROMATE SOLUTION, ML.
0 to 1	10
1 to 2	15
2 to 3	20
3 to 4	25
4 to 5	30
5 to 6	35

PHOSPHORUS

Special Solutions Required

5. (*a*) *Ammonium Molybdate.*—Dissolve 100 g. of molybdic acid (85 per cent MoO_3) in a mixture of 400 ml. of water and 100 ml. of NH_4OH (sp. gr. 0.90) and cool the solution to room temperature. While stirring constantly, pour this solution very slowly into a cooled mixture of 500 ml. of water and 400 ml. of HNO_3 (sp. gr. 1.42). No permanent precipitate should form, which does not redissolve with stirring. Add 1 drop of a saturated solution of sodium-ammonium phosphate ($NaNH_4HPO_4$) to the solution, shake it thoroughly, and allow it to stand at least 24 hr. before use. Filter portions of the solution as needed.

(*b*) *Magnesia Mixture.*—Dissolve 50 g. of $MgCl_2 \cdot 6H_2O$ and 100 g. of NH_4Cl in 500 ml. of water. Make the solution slightly alkaline with NH_4OH and allow it to stand at least 12 hr. Filter the solution, acidify it with HCl and dilute to 1 liter.

Procedure

6. (*a*) Weigh a 1 to 3-g. sample of cement (Note) into a 250-ml. beaker Treat the cement with 50 to 75 ml. of water and then with 10 to 15 ml. of HNO_3 (sp. gr. 1.42). Boil the mixture until the solution is as complete as possible. If the cement contains an appreciable amount of manganese as shown by the presence of a red or brown residue, add a few milliliters of a potassium nitrite solution (5 per cent) to aid in the solution. Boil the solution until all nitrous fumes are completely expelled, taking care not to allow the volume of the solution to become so small as to cause the precipitation of gelatinous silica. Filter the solution into a 500-ml. Erlenmeyer flask and wash the residue. The volume of the solution should now be about 150 ml.

NOTE.—The amount of sample and reagents used depend on the content of phosphorus in the cement. The minimum requirements are sufficient if the cement contains 0.5 per cent P_2O_5 or more. The maximum amounts are required if the content of P_2O_5 is 0.1 per cent or less.

(*b*) Add 10 g. of ammonium nitrate to the solution and heat until the salt is dissolved. Heat the solution to 85 C. and add 50 to 100 ml. of the solution of ammonium molybdate. Stopper the flask and shake it vigorously for 5 min., then set it aside for 30 min. Filter the precipitate, rinsing the flask five times and washing the filter paper eight times with a cold solution of potassium nitrate (1 per cent).

(*c*) Put the original flask under the funnel containing the precipitate. Pour 25 ml. of diluted NH_4OH (1:4) over the precipitate and around the upper edges of the filter paper until the precipitate is entirely dissolved. Wash the paper three times with cold water, twice with diluted HCl (1:1), and finally three times with cold water. Acidify the solution with HCl and add to it 4 or 5 drops of a saturated solution of citric acid to hold traces of iron, if any, in solution. Cool the solution to room temperature and transfer it to a 400-ml. beaker. The volume of the solution should now be about 150 ml.

(*d*) To the solution add 25 ml. of magnesia mixture, and then while stirring constantly add drop by drop 50 ml. of NH_4OH (sp. gr. 0.90) at such a rate that 5 min. are required for the addition. Allow the mixture to stand 4 hr. or longer. Filter the precipitate and wash it a few times with diluted NH_4OH (1:20). Dissolve the precipitate into the original beaker by pouring 25 ml. of diluted HCl (1:1) on the precipitate. Wash the filter paper thoroughly with diluted HCl (1:20). With the washings and water, dilute the solution to 50 to 100 ml. and add to it 1 to 2 ml. of magnesia mixture. While stirring constantly, add NH_4OH slowly to the solution as before. Allow the mixture to stand 2 hr. or longer. Filter the precipitate, scrub and rinse the beaker, and wash the precipitate ten times with diluted NH_4OH (1:20). Ignite the precipitate in a weighed crucible at 1100 to 1200 C. to constant weight. Multiply the weight of the magnesium pyrophosphate ($Mg_2P_2O_7$) by 0.6379 to get the weight of the phosphorus in the sample as P_2O_5.

(*e*) *Blank Correction.*—Make a blank determination, using the same procedure as outlined above, and using approximately the same amount of reagents. Correct the weight of magnesium pyrophosphate ($Mg_2P_2O_7$) accordingly.

(*f*) If a determination of phosphorus is required, the weight of the ferric oxide and alumina ($Al_2O_3 + Fe_2O_3$) as obtained by the method described in Section 12 of the Standard Methods of Sampling and Testing Portland Cement (A.S.T.M. Designation: C 77) of the American Society for Testing Materals[1] shall be corrected for the amount of the P_2O_5 found.

MANGANESE

Special Solutions Required

7. (*a*) *Standard Sodium Arsenite Solution.*—Dissolve in 100 ml. of water 3.0 g. of Na_2CO_3 and then 0.9008 g. of As_2O_3, heating the mixture until the solution is as complete as possible. If the solution is not clear or contains a residue, filter the solution. Cool it to room temperature, transfer it to a volumetric flask, and dilute it to 1 liter. Each milliliter of this solution is approximately equivalent to 0.003 g. of Mn_2O_3.

(*b*) Dissolve 0.58 g. of $KMnO_4$ in 1 liter of water and standardize it against 0.0300 g. of sodium oxalate oxidimetric standard furnished by the National Bureau of Standards (Standard Sample No. 40) according to the directions furnished with the sodium oxalate. Put 30.0 ml. of the $KMnO_4$ solution in a 250-ml. Erlenmeyer flask. Add 60 ml. of diluted HNO_3 (1:4) and 10 ml. of a potassium nitrite solution (5 per cent) to the flask. Boil the solution until the nitrous acid is completely expelled. Cool the solution and add sodium bismuthate and finish by titrating with the standard solution of As_2O_3 in the manner described below for the determination of manganese in cement.

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[1] 1937 Supplement to Book of A.S.T.M. Standards, p. 54.

(*c*) The value of the standard solution of As_2O_3 in terms of Mn_2O_3 is:

$$\frac{0.03 \times 0.236 \times 30}{a \times b}$$

where a = the number of milliliters of the solution of $KMnO_4$ required by 0.0300 g. of $Na_2C_2O_4$, and

b = the number of milliliters of the standard solution of As_2O_3 required by 30.0 ml. of the solution of $KMnO_4$.

Procedure

8. (*a*) Weigh a 1.0 to 3.0-g. sample of cement (Note 1) into a 250-ml. beaker and treat it with 5 to 10 ml. of water and then with 60 to 75 ml. of diluted HNO_3 (1:4). Boil the mixture until the solution is as complete as possible. Add 10 ml. of a sodium nitrite solution (5 per cent) to the solution and boil it until the nitrous acid is completely expelled (Note 2), taking care not to allow the volume of the solution to become so small as to cause the precipitation of a gelatinous silica. There may be some separated silica which may be ignored; but if there is still a red or brown residue, use more solution of sodium nitrite to effect a complete decomposition and then boil again to expel the nitrous acid. Filter the solution into a 250-ml. Erlenmeyer flask and wash the filter paper with water.

NOTE 1.—The amount of cement taken for analysis depends on the content of manganese, varying from 1 g. for about 1 per cent of Mn_2O_3 to 3 g. for 0.25 per cent or less of Mn_2O_3.

NOTE 2.—When sodium nitrite is added, the expulsion of nitrous acid by boiling must be complete. If any nitrous acid remains in the solution, it will react with the added sodium bismuthate and decrease its oxidizing value. If there is any manganese in the cement, the first small quantity of sodium bismuthate should bring out a purple color.

(*b*) The solution should have a volume of 100 to 125 ml. Cool it to room temperature. To the solution add a total of 0.5 g. of sodium bismuthate in small quantities, with intermittent shaking. After the addition is completed, shake the solution occasionally for 5 min. and then add to it 50 ml. of cool diluted HNO_3 (1:33) which has been previously boiled to expel nitrous acid. Filter the solution through a pad of ignited asbestos in a Gooch crucible or a carbon filter with the aid of suction. Wash the residue four times with the cool diluted HNO_3 (1:33). Titrate the filtrate immediately with the standard solution of As_2O_3. The endpoint is reached when a yellow color is obtained free of brown or purple tints and it does not change upon further addition of As_2O_3. Calculate the manganese in the cement as Mn_2O_3.

(*c*) *Blank Correction.*—Make a blank determination, using the same procedure as outlined above and using approximately the same amounts of reagents. Correct the titration results accordingly.

(*d*) If a determination of manganese is required in addition to a determination of magnesium by Section 16 of Standard Method C 77,[1] manganese shall be eliminated from the combined filtrates obtained in the alumina and ferric oxide determination before calcium oxide is precipitated (Section 15 of Standard Method C 77)[1] in the following manner:

Acidify the combined filtrates with HCl and evaporate them to a volume of about 100 ml. Add 40 ml. of saturated bromine water to the hot solution and immediately add NH_4OH until the solution is distinctly alkaline. Boil the solution for 5 min. or more, making certain that the solution is at all times distinctly alkaline. Allow the precipitate to settle, filter and wash with hot water. Discard the precipitate. Acidify the filtrate with HCl and boil until all the bromine is expelled. Make the solution alkaline with NH_4OH and proceed in accordance with Section 15 of Standard Method C 77.[1]

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[1] 1937 Supplement to Book of A.S.T.M. Standards, p. 54.

TENTATIVE METHOD OF TEST
FOR
COMPRESSIVE STRENGTH OF PORTLAND-CEMENT MORTARS[1]

A.S.T.M. Designation: C 109 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1934; REVISED, 1937.

Scope

1. This method of test is intended for determining the compressive strength of portland-cement mortars.

APPARATUS

Scales

2. (*a*) The scales used in weighing materials for mortar mixes shall conform to the following requirements: On scales in use the tolerance at a load of 1500 g. shall be ± 1.0 g. The tolerance on new scales shall be one-half of this value. The "sensibility reciprocal"[2] shall not be greater than twice the tolerance.

(*b*) The tolerances on the weights in use in weighing materials for mortar mixes shall be as follows:

WEIGHT	TOLERANCE	WEIGHT	TOLERANCE
1000 g.	±0.5 g.	100 g.	±0.15 g.
900 g.	±0.45 g.	50 g.	±0.10 g.
750 g.	±0.4 g.	20 g.	±0.05 g.
500 g.	±0.35 g.	10 g.	±0.04 g.
300 g.	±0.3 g.	5 g.	±0.03 g.
250 g.	±0.25 g.	2 g.	±0.02 g.
200 g.	±0.20 g.	1 g.	±0.01 g.

The tolerances on new weights shall be one-half these values.

Glass Graduates

3. Glass graduates of 250-ml. capacities used for measuring the mixing water shall have the main graduation lines completely encircling the

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] The sensibility reciprocal is a measure of the sensitivity of a balance, and is the weight required to move the position of equilibrium of the beam, pan, pointer, or other indicating device of a scale a definite amount at the capacity or at any lesser load. For a complete definition of sensibility reciprocal, see National Bureau of Standards *Handbook, M 85*, pp. 80–82.

cylinder and the intermediate lines at least half-way around the cylinder and shall be made to deliver the indicated volume at 20 C. (68 F.). The tolerance on these graduates shall be ±2.0 ml.

Molds

4. (*a*) Molds for the 2-in. cube test specimens shall be tight fitting. The parts of the molds when assembled shall be positively held together. The molds shall be made of hard metal not attacked by the cement mortar. For new molds the Rockwell hardness number of the metal shall be not less than B55 (the Brinell hardness number not less than 95). There shall be sufficient material in the sides of the molds to prevent spreading or warping.

(*b*) The interior faces of the molds shall be true plane surfaces with a tolerance of 0.001 in. for new molds or 0.002 in. for molds in use.

(*c*) The distances between opposite faces shall be 2 in. ± 0.005 in. for new molds, or 2 in. ± 0.01 in. for molds in use. The height of the molds, measured separately for each cube compartment, shall be 2 in. with tolerances of +0.01 in. and −0.005 in. for new molds; or +0.01 in. and −0.015 in. for molds in use.

(*d*) The angle between adjacent interior faces, and between interior faces and top and bottom planes of the mold shall be 90 deg. ± 0.5 deg.

Testing Machine

5. (*a*) The testing machine for determination of the compressive strength may be either the hydraulic or the screw-type with sufficient opening between the upper bearing surface and the lower bearing surface of the machine to permit the use of verifying apparatus. The load applied to the test specimen shall be indicated with an accuracy of ±1.0 per cent.

(*b*) The upper bearing shall be a spherically seated, hardened, metal block firmly attached at the center of the upper head of the machine. The center of the sphere shall lie at the center of the surface of the block in contact with the specimen. The block shall be closely held in its spherical seat, but shall be free to turn in any direction. The diagonal or diameter[1] of the bearing surface shall be only slightly greater than the diagonal of the face of the 2-in. cube in order to facilitate accurate centering of the specimen. A hardened metal bearing block shall be used beneath the specimen to minimize wear of the lower platen of the machine. The bearing block surfaces intended for contact with the specimen should have a hardness not less than C60 Rockwell number (Brinell number 620). These surfaces shall not depart from plane surfaces by more than 0.0005 in. when the blocks are new and shall be maintained within a tolerance of 0.001 in.

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[1] A diameter of $3\frac{1}{8}$ in., which is large enough for testing 3 by 6-in. cylinders, is satisfactory provided that the lower bearing block has a diameter slightly greater than the diagonal of the face of the 2-in. cube but not more than 2.9 in., and is centered with respect to the upper bearing block and is held in position by suitable means.

Temperature and Humidity

Temperature and Humidity

6. The temperature of the room and dry materials shall be maintained at not less than 20 C. (68 F.) and not more than 27.5 C. (81.5 F.). The temperature of the mixing water, moist closet, and water in the storage tank shall not vary from 21 C. (70 F.) more than 1.7 C. (3 F.). The moist closet or moist room shall be so constructed as to provide storage facilities for test specimens at a relative humidity of not less than 90 per cent.

Preparation of Specimens

Graded Ottawa Sand

7. The sand (Note 1) shall be natural silica sand produced by the Ottawa Silica Co., Ottawa, Ill., graded as follows:

Retained on No. 100 sieve	98 ± 2 per cent
Retained on No. 50 sieve	72 ± 5 per cent
Retained on No. 30 sieve	2 ± 2 per cent
Retained on No. 16 sieve	0 per cent

Sieve Analysis of Sand

8. (*a*) For checking the grading of the sand, a sieving test shall be made on each of the four sieves specified in Section 7. Samples for these tests shall be quartered from a sample of about 700 g. obtained by the method of quartering the contents of a full sack (100 lb.) which have been thoroughly mixed and the pile flattened or spread out to minimize segregation during quartering.

(*b*) The test on each sieve shall be made with 100 g. of sand. The sand shall be placed on the sieve, which shall be thoroughly clean and dry. The sieve, with pan and cover attached, shall be held in one hand in a slightly inclined position so that the sample will be well distributed over the sieve, at the same time gently striking the side about 150 times per minute against the palm of the other hand on the up stroke. The sieve shall be turned every 25 strokes about one-sixth of a revolution in the same direction. The operation shall be continued until not more than 0.5 g. passes through in 1 min. of continuous sieving. The weight of the residue on the sieve shall be expressed as a percentage of the weight of the original sample.

Sieve Requirements

9. The Nos. 100, 50, 30 and 16 sieves shall conform to the requirements for these sieves as specified in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[1]

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

Preparing Molds

10. Molds shall be thinly covered with mineral oil. The clamped faces shall be thinly covered with a heavy mineral oil or light cup grease. After assembling the mold, excess oil or grease shall be wiped from the joints. Molds shall be set on a plane, non-absorbent base plate which has been thinly coated with paraffin or mineral oil. A mixture of 3 parts paraffin to 5 parts resin by weight heated to between 110 and 120 C. (230 and 248 F.) shall be applied at the outside contact line of the mold and base plate so that a water-tight bond is effected between mold and base (Note 2).

Proportioning and Mixing of Mortars

11. (*a*) The proportions of the standard mortar shall be 1 part cement to 2.77 parts graded Ottawa sand by weight and 53 per cent water by weight of cement (water-cement ratio of 0.8 by volume). The standard batch of mortar shall consist of 470 g. of cement, 1300 g. of graded Ottawa sand and 250 ml. of water.

(*b*) Mixing shall be done in a bowl (enamelware or other non-absorbent, non-corroding material) of about 1-gal. capacity by vigorous and continuous stirring, squeezing and kneading with one hand which shall be protected by a snug-fitting rubber glove. The materials, sufficient for making six cubes, shall be introduced in the following manner:

(*1*) Place the water in the bowl, which has just been wiped with a damp cloth,
(*2*) Add the cement to the water, and mix for 30 sec.,
(*3*) Add approximately one-half of the testing sand, and mix for 30 sec., and
(*4*) Add the remainder of the sand, and mix for 1½ min.

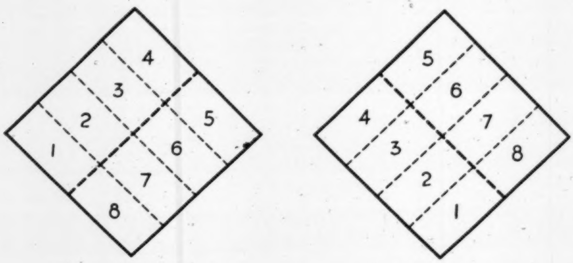
Molding Specimens

12. Immediately following completion of the mixing, the mortar adhering to the mixing glove shall be removed by striking the hand against the side or edge of the mixing bowl and the glove shall then be removed. A layer of mortar about 1 in. in thickness shall be placed in each of the six cubes. The mortar in each cube shall be tamped 32 times in about 10 sec. in four rounds, each round to be at right angles to the other and consisting of eight adjoining strokes over the surface of the specimen, as illustrated in Fig. 1, applying to the mortar on each stroke a pressure of about 150 g. (Note 5). The tamper shall have a cross-section of ½ by 1 in., it shall be of a convenient length (5 or 6 in.) and made of a non-absorptive, non-abrasive material such as medium hard rubber. The four rounds of tamping (32 strokes) of the mortar shall be completed in one cube before going to the next. When the tamping of the first layer in the six cubes is completed, the cubes shall be filled with the remaining mortar

and again tamped as specified for the first layer. On completion of the tamping, the tops of all cubes should extend slightly above the top surface of the molds. The mortar which has been forced out onto the top surface of the mold shall be brought in with a trowel and the cubes smoothed off by drawing the flat side of the trowel once across the top of each cube at right angles to the length of the mold. The mortar shall then be cut off to a level surface flush with the top of the mold by drawing the straight edge of the trowel with a sawing motion over the length of the mold.

Storage of Test Specimens

13. Immediately after molding, the specimens shall be placed in the moist closet or moist room with their upper surfaces exposed to the moist air but protected from dripping water. Specimens shall remain in the molds for at least 20 hr.; if removed from the molds before 24 hr., they shall be kept on shelves of the moist closet or moist room until expiration of the 24-hr. period. When 24 hr. old, the specimens shall be immersed in clean water in storage tanks constructed of non-corroding materials. The storage water shall be kept clean by frequent changing.



Rounds 1 and 3 Rounds 2 and 4

FIG. 1.—Order of Tamping in Molding of Test Specimens.

Testing of Cubes

14. (*a*) Testing of the cube specimens shall be carried out immediately after their removal from the moist closet for 24-hr. specimens, and from storage water for all other specimens. If more than one specimen at a time is removed from the storage water for testing, these cubes shall be placed in a pan of water of sufficient depth to completely immerse each cube until its removal for testing. The load shall be applied to the faces of the cubes that were in contact with the true plane surfaces of the mold. These faces shall be checked by application of an accurate straight edge (Note 3). If appreciable curvature is present, the face or faces shall be ground to a plane surface before loading, or the specimens shall be discarded.

(*b*) Loose sand grains or incrustations shall be removed from the contact faces and cubes shall then be carefully placed in the testing machine below the center of the upper bearing block. No cushioning or bedding materials shall be used. The loading up to 50 per cent (25 per cent for expected maximum loads of less than 4000 lb. at ages of 7 days or less) of the expected maximum load may be applied at any convenient rate, after which the specimens shall be loaded continuously to failure at a rate or rates which shall at no time be less than 1000 nor more than 6000 lb. per sq. in. per min.[1]

Calculation of Strength

15. The total maximum load indicated by the testing machine shall be recorded, and the compressive strength calculated in pounds per square inch from the cross-sectional area of the cube tested (Note 4). Cubes that are manifestly faulty or that give strengths differing more than 10 per cent from the average value of all test specimens made from the same sample and tested at the same period shall not be considered in determining the compressive strength (Note 5).

EXPLANATORY NOTES

NOTE 1.—*Segregation of Graded Sand:* The graded Ottawa sand should be handled in such a manner as to prevent segregation, since variations in the grading of the sand cause variations in the consistency of the mortar. It is recommended that each sack of sand be emptied when delivered to the laboratory for use and its contents mixed by means of a shovel or scoop. The sand may then be resacked if desired and scooped directly from the sack as needed in testing. In both of these operations care should be taken to prevent segregation. In emptying sacks of sand into bins or in scooping it out of bins, care should be exercised to prevent the formation of mounds of sand or craters in the sand, down the slopes of which the coarser particles will roll. Bins should be of sufficient size to permit the above precautions. Devices for drawing the sand from bins by gravity should not be used.

NOTE 2.—*Water-tight Molds:* The mixture of paraffin and resin specified for sealing the joint between mold and base plate may be found difficult to remove when molds are being cleaned. A water-tight joint may be secured with paraffin alone by slightly warming the mold and base plate before brushing the joint. Molds so treated should be allowed to return to room temperature before use. Use of straight paraffin is permissible *if a water-tight joint is secured.*

NOTE 3.—*Cube Faces:* Results much lower than the true strength will be obtained by loading faces of the cube which are not truly plane surfaces. Therefore, it is essential that cube molds be kept scrupulously clean, as otherwise large irregularities in the surfaces will occur. Instruments for cleaning of molds should always be softer than the metal in the molds to prevent wear. In case grinding of cube faces is necessary, it can be accomplished best by rubbing the cube on a sheet of fine emery paper or cloth glued to a plane surface, using only a moderate pressure. Such grinding is tedious for more than a few thousandths of an inch; where more than this is found necessary, it is recommended that the cube be discarded.

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[1] While the data presented in the report of the 1933 cooperative tests (see the Report of Working Committee on Plastic Mortar Tests for Portland Cement, of Committee C-1 on Cement, *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 322 (1934)) do not give conclusive evidence on effect of rate of loading, the Working Committee favors the use of a uniform rate of loading of about 4000 lb. per sq. in. per min. (1000 lb. per sq. in. per min. for low-heat portland cements at ages of 7 days or less).

NOTE 4.—*Calculation of Strength:* Ordinarily, the cross-sectional area of the cube tested may be assumed to be 4.00 sq. in.; however, in cases where the area of the cube varies more than 0.06 sq. in. from 4.00 sq. in., the actual area shall be used for calculation of the compressive strength.

NOTE 5.—*Faulty Cubes:* Faulty cubes may result from occlusions of air during molding unless care is exercised. All indentations resulting from the puddling of the lower layer of mortar should be smoothed out before introducing the top layer. Excess puddling, resulting in segregation of the materials, should be avoided. In testing, cubes should be loaded to destruction and after rupture their centers examined for air pockets. Improper centering of cubes resulting in oblique fractures or lateral movement of one of the heads of the machine during loading will often cause lower strength results. Cubes so broken shall be considered as "manifestly faulty" unless their strengths agree within reasonable limits with those of cubes exhibiting a normal pyramidal fracture.

TENTATIVE SPECIFICATIONS
FOR
BUILDING BRICK (MADE FROM CLAY OR SHALE)[1]

A.S.T.M. Designation: C 62 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society **is subject to** annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1935; REVISED, 1936, 1937.

Scope

1. (*a*) These specifications cover brick made from clay or shale and burned, and which are intended for use in brick masonry. Three grades of brick are covered, as follows:

Grade SW.—Bricks intended for use where exposed to temperature below freezing in the presence of moisture.

NOTE.—As a typical example, bricks used for foundation courses and parapets in the northeastern quarter of the United States should conform to this grade.

Grade MW.—Bricks intended for use where exposed to temperature below freezing but unlikely to be saturated with water.

NOTE.—As a typical example, bricks exposed in the face of the wall other than parapet or foundations, or bricks intended for structures located in regions of the United States characterized by less severe frost action or by drier climate than is found in the northeastern quarter of the United States, should conform to this grade.

Grade NW.—Bricks intended for use as back-up or interior masonry, or if exposed, for use where no frost action occurs; or if frost action occurs where the average annual precipitation is less than 15 in.

(*b*) Where bricks having strengths greater than called for by these specifications are required, the purchaser should specify the desired minimum compressive strength.

NOTE.—See "Modifications in Recommended Minimum Requirements for Masonry Wall Construction," Report of Building Code Committee of U. S. Department of Commerce (1931). In this report the following four grades of brick according to compressive strength, tested flatwise, are recognized: 1500 to 2500, 2500 to 4500, 4500 to 8000, and over 8000 lb. per sq. in.

Properties

2. (*a*) The bricks shall conform to the physical requirements for the grade specified, as follows:

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

These Tentative Specifications are in effect a tentative revision of, and are intended to supersede when adopted as standard, the present Standard Specifications for Building Brick (Made from Clay or Shale) (A.S.T.M. Designation: C 62 - 30), 1936 Book of A.S.T.M. Standards, Part II, p. 119.

	Compressive Strength (bricks flatwise), lb. per sq. in., average gross area		Water Absorption by 5-hr. Boiling, per cent		C/B Ratio[a]	
	Average of 5 Bricks	Individual Minimum	Average of 5 Bricks	Individual Maximum	Average of 5 Bricks	Individual Maximum
Grade *SW*	2500	2500	20	20	0.80	0.80
Grade *MW*	2500	2000	20	25	0.85	0.90
Grade *NW*	1500	1250	no limit	no limit	no limit	no limit

[a] The ratio *C/B* is the ratio of absorption by 24-hr. submersion in cold water to that after 5-hr. submersion in boiling water.

(*b*) Unless otherwise specified by the purchaser, bricks of Grades *SW* and *MW* shall be accepted in lieu of Grade *NW*, and Grade *SW* in lieu of Grade *MW*.

(*c*) If the average compressive strength is greater than 8000 lb. per sq. in. *or* the average water absorption is less than 8 per cent by weight after 5-hr. submersion in boiling water, *or* less than 6.4 per cent after 24-hr. submersion in cold water, the requirement for *C/B* ratio shall be waived.

Test Specimens

3. For purpose of tests, bricks representative of the commercial product shall be selected by a competent person appointed by the purchaser, the place or places of selection to be designated when the purchase order is placed. The manufacturer or seller shall furnish specimens for tests without charge. All bricks shall be carefully examined and their condition noted before testing. For the purpose of tests, not less than five brick shall be required for each investigation. In general, two samples of five brick each shall be tested for each 100,000 bricks contained in the lot under consideration; but where the total quantity exceeds, 500,000, one sample of five brick from each 100,000 shall be tested. Additional representative samples may be taken at any time or place at the discretion of the purchaser.

Standard Sizes

4. The standard sizes of brick made from clay or shale shall conform to the following dimensions, within permissible variations of $\pm \frac{1}{16}$ in. in depth, $\pm \frac{1}{8}$ in. in width, and $\pm \frac{1}{4}$ in. in length:

TYPE	DEPTH, IN.	WIDTH, IN.	LENGTH, IN.
Common brick	$2\frac{1}{4}$	$3\frac{3}{4}$	8
Rough-face brick	$2\frac{1}{4}$	$3\frac{3}{4}$	8
Smooth-face brick	$2\frac{1}{4}$	$3\frac{7}{8}$	8

Workmanship and Finish

5. When any particular surface appearance or uniformity is desired, it shall be as mutually agreed upon by the manufacturer and the purchaser.

Visual Inspection

6. All brick shall be free from cracks, warpage, stones, pebbles, or particles of lime that would affect their serviceability or strength.

Methods of Testing

7. The bricks shall be tested in accordance with the Standard Methods of Testing Brick (Compressive Strength and Absorption) (A.S.T.M. Designation: C 67) of the American Society for Testing Materials.[1]

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[1] 1937 Supplement to Book of A.S.T.M. Standards, p. 78.

TENTATIVE SPECIFICATIONS
FOR
GLAZED BUILDING UNITS[1]

A.S.T.M. Designation: C 126 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1936; REVISED, 1937.[2]

Scope

1. These specifications cover brick, tile, and other masonry building units of clay or shale with a finish consisting of a vitreous glaze, excluding natural salt glazed ware.

Sizes

2. (*a*) The units shall be of the sizes and shapes specified (see Explanatory Note 1), no dimension differing by more than 2 per cent from that specified.

(*b*) The units may be either solid or cored, but the area of the cells shall not exceed 40 per cent of the gross area of a section perpendicular to the axis of the cells. The area of the cells in units intended for use where subjected to impact shall not exceed 25 per cent of the gross sectional area.

Workmanship

3. (*a*) All units shall be straight with sharp edges and corners, unless otherwise specified. Faces intended to be plane shall have no distortions greater than 1 per cent of the larger dimension of the face.

(*b*) All surfaces that will be exposed when the units are in place shall be finished with a glaze of uniform quality. The glaze shall be free from chips, crazes, blisters, crawling and other imperfections.

Finish

4. The texture and color of the finish shall be as specified (see Explanatory Note 2).

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Revision accepted by Committee E-10 on Standards, August 26, 1937.

Imperviousness of Finish

5. No trace of the stain shall remain on the finish after the staining test, except that, if a finish with a matt texture is specified, a slight discoloration in depressions will not be cause for rejection.

Opacity of Finish

6. When an opaque glaze is specified no penetration of the stain shall be visible through the finish within 5 min. after the staining solution has been applied in the opacity test.

Acid Resistance of Finish

7. Neither the texture nor the color of the finish shall be altered by the exposure in the acid resistance test.[1]

Resistance to Crazing

8. The finish shall not craze, spall, peel, or crack during the exposure in the autoclave test.

Absorption of Body

9. When the units are intended for use where exposed to frost action, the absorption of the body of the unit by 24-hr. submersion in cold water shall not exceed 14 per cent, and *in addition* the units shall conform to at least *one* of the following requirements for absorption:

(*a*) The absorption by 24-hr. submersion in cold water shall not exceed 7 per cent.

(*b*) The ratio of the absorption by 24-hr. submersion in water to the absorption by 5-hr. submersion in boiling water shall not exceed 0.80.

Inspection

10. Facilities shall be provided the purchaser for sampling and inspection at either the plant or the site of the construction, as specified by the purchaser. At least 5 days from the time of sampling shall be allowed for the completion of the tests.

Rejection

11. At the option of the purchaser, a lot of units may be rejected if it fails to meet any of the requirements of these specifications.

SAMPLING AND METHODS OF TESTING

Sampling

12. (*a*) Specimens of whole building units shall be selected by the purchaser or his representative at the place or places designated in the purchase order.

(*b*) Five specimens shall be tested for a lot of 10,000 units or fraction thereof; for larger lots 5 additional specimens shall be tested for each 30,000 units or fraction thereof.

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[1] Intended as a measure of resistance of colored finishes to fading.

(*c*) Other samples may be taken at any time or place at the discretion of the purchaser.

(*d*) The manufacturer or seller shall furnish for testing, without charge, a minimum of 10 units and additional specimens up to 0.1 per cent of each size and finish in the lot.

Imperviousness of Finish Test

13. Liquid ink or methylene blue shall be applied liberally to the finish, and allowed to remain for 5 min. The surface shall then be washed with a wet cloth and running water, and examined for staining of the finish.

Opacity Test of Finish

14. Liquid ink or methylene blue shall be applied to the body of dry specimens of glazed building units at an edge of a finished surface. After 5 min. the finish shall be examined for opacity.

Acid Resistance Test of Finish

15. A portion of the glazed surface of one or more of the specimens of each texture and color shall be submerged in a 10 per cent solution of hydrochloric acid at a temperature of 60 to 80 F. (15 to 27 C.) for 3 hr. The finish shall then be rinsed, dried, and examined for changes of texture and color.

Resistance to Crazing Test
(Autoclave Test)

16. Dry specimens, previously tested for imperviousness of finish (Section 13), shall be placed above the water in an autoclave. Units which have been tested also for opacity (Section 14) and acid resistance of finish (Section 15) may be used. The pressure within the autoclave shall be increased gradually by admitting saturated steam or by heating the water, the air being allowed to escape until substantially all of the air has been replaced by steam. The pressure shall be increased to between 20 and 50 lb. per sq. in. in 30 min., to between 60 and 100 lb. per sq. in. in 60 min., and to 150 lb. per sq. in. in from 80 to 120 min. after the beginning of the steaming. The pressure shall be held at 150 ± 5 lb. per sq. in. for 1 hr. The pressure shall then be released in a period of from 10 to 30 min. and the specimens then allowed to cool gradually to room temperature. A record of either the temperature or the pressure within the autoclave shall be maintained, including values of either temperature or pressure at intervals not to exceed 10 min. during the test. Ink of a color differing from that of the stain used in the test for imperviousness of finish shall then be applied to the finish and allowed to stand for 5 min. The surface shall then be washed with a wet cloth and running water, and examined for crazing of the glaze.

Absorption Test

17. The absorption tests shall be made in accordance with the Standard Methods of Testing Brick (Modulus of Rupture, Compressive Strength, Absorption) (A.S.T.M. Designation: C 67) of the American Society for Testing Materials.[1]

EXPLANATORY NOTES

NOTE 1.—The following sizes and shapes are standard in the industry:

HEIGHT, IN.	LENGTH, IN.	THICKNESS, IN.
$2\frac{1}{4}$	8	$1\frac{3}{4}$, $1\frac{7}{8}$, or $3\frac{7}{8}$
$3\frac{7}{8}$	8	$1\frac{3}{4}$, $1\frac{7}{8}$, or $3\frac{7}{8}$
5	8	$1\frac{3}{4}$, $1\frac{7}{8}$, or $3\frac{7}{8}$
5	10	$1\frac{3}{4}$, $1\frac{7}{8}$, or $3\frac{7}{8}$
5	12	$1\frac{3}{4}$, $1\frac{7}{8}$, or $3\frac{7}{8}$
6	12	$1\frac{3}{4}$, $1\frac{7}{8}$, or $3\frac{7}{8}$
$7\frac{7}{8}$	$7\frac{7}{8}$	$1\frac{3}{4}$, $1\frac{7}{8}$, or $3\frac{7}{8}$
$7\frac{7}{8}$	16	$1\frac{3}{4}$, $1\frac{7}{8}$, or $3\frac{7}{8}$
$10\frac{1}{4}$	12	$1\frac{3}{4}$, $1\frac{7}{8}$, or $3\frac{7}{8}$
12	12	$1\frac{3}{4}$, $1\frac{7}{8}$, or $3\frac{7}{8}$

NOTE 2.—As an aid to the designer, the textures and colors listed below are standard in the industry. All colors and textures, however, are not available in all areas; additional colors and textures are available in some markets. It is suggested, therefore, that the designer inquire into the colors and textures available in the locality where the product is to be used.

CHART OF TEXTURES AND COLORS OF GLAZED BUILDING UNITS STANDARD IN THE INDUSTRY

Textures		Colors		
Ceramic Glazed	Coated Glaze	Glazed	Ceramic Glazes	
			Light Shades	*Trim Shades*
Lustrous Gloss Satin Matt Porcelain	Gloss	Gray Cream Light buff Medium buff Dark buff Orange Maroon Blends of Grays and Creams Creams and Light Buffs Light and Medium Buffs Medium and Dark Buffs Full Range	White Cream Ivory Buff Tan Gray Various Light Speckles Stipples or Ripples of the above light colors Mottles of two or more of the above light colors Mottles of light and trim colors, predominatingly of light colors	Black Browns Blues Greens Reds Yellow Gold Stipples, Ripples, or Mottles of the above dark colors Mottles of trim and light colors, predominatingly dark

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[1] 1937 Supplement to Book of A.S.T.M. Standards, p. 78.

TENTATIVE SPECIFICATIONS
FOR
PAVING BRICK[1]

A.S.T.M. Designation: C 7 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1913; ADOPTED, 1915; REVISED, 1930; REISSUED AS TENTATIVE, 1937; REVISED, 1937.[2]

Scope

1. These specifications cover vitrified brick used in the construction of pavements.

MANUFACTURE

General Requirements

2. Paving brick shall be manufactured from fire clay, semi-fire clay or shale, or combinations thereof. The manufacturing process shall be such that the brick are thoroughly annealed, tough, durable and evenly burned so that, when broken, they show a uniformly dense structure free from lime, air pockets and marked laminations. The name of the brick or the manufacturer shall be impressed on the side or end of each brick in recessed letters.

Side Lugs

3. The brick shall be provided on one side with not less than two lugs, which shall be not less than $\frac{1}{8}$ in. nor more than $\frac{1}{4}$ in. in height. The area of the projections or lugs measured at the base of the lug shall not exceed in total area 3 sq. in. No lug shall be continuous from top to bottom of the brick, but shall be of a type that allows free horizontal flow of the filler.

End Lugs

4. Vertical fiber brick shall be provided with not less than two non-continuous lugs or portions of lugs on each end of each brick. Lugs shall be non-meshing and shall have a total base area of not more than $2\frac{1}{2}$ sq. in. and shall be manufactured so there may be free horizontal flow of the filler.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

These specifications are in effect a revision of and replace the former Standard Specifications for Paving Brick (C 7 – 30), which standard was accordingly discontinued in 1937.

[2] Latest revision accepted by Committee E-10 on Standards, August 26, 1937.

The lugs shall project from the ends of the brick so as to insure an end joint of not less than $\frac{1}{8}$ in. nor more than $\frac{1}{4}$ in. in width.

Types and Sizes

5. Paving brick shall conform to the following types and nominal sizes. The dimensions of the brick shall be construed as applying to the body of the brick only, exclusive of lugs.

	DEPTH, IN.	WIDTH, IN.	LENGTH, IN.
Repressed Lug Brick	4	$3\frac{1}{2}$	$8\frac{1}{2}$
Vertical Fiber Lug Brick	$2\frac{1}{2}$	4	$8\frac{1}{2}$
	3	4	$8\frac{1}{2}$
	$3\frac{1}{2}$	4	$8\frac{1}{2}$

NOTE.—The above sizes and varieties are those recognized by the Permanent Committee on Simplification of Varieties and Standards for Vitrified Paving Brick, Simplified Practice Recommendation R 1–36, of the U. S. Department of Commerce, for the years 1936 and 1937, and are subject to change from year to year.

Repressed Brick

6. The edges of repressed brick shall be rounded to a radius of not more than $\frac{3}{16}$ in.

Permissible Variations in Size

7. For any lot of paving brick furnished under these specifications, the individual bricks shall not vary from the nominal size requirements specified in Section 5 by more than $\pm\frac{1}{8}$ in. in either transverse dimension, exclusive of kiln marks, or by more than $\pm\frac{1}{4}$ in. in length.

PHYSICAL PROPERTIES AND TESTS

Loss in Rattler Test

8. (*a*) The percentage of loss in the rattler test of the respective sizes of paving brick specified shall not exceed the following maximum limits:

SIZE OF BRICK		MAXIMUM LOSS IN RATTLER TEST, PER CENT
TRANSVERSE DIMENSIONS, IN.	LENGTH, IN.	
$2\frac{1}{2}$ by 4	$8\frac{1}{2}$	26
3 by 4	$8\frac{1}{2}$	24
$3\frac{1}{2}$ by 4	$8\frac{1}{2}$	22

(*b*) The total number of pieces weighing 1 lb. or more remaining after the completion of the rattler test shall not exceed 12.

VISUAL INSPECTION

Rejection by Visual Inspection

9. Paving brick shall be subject to visual inspection prior to shipment, after delivery, and during laying for the purpose of culling out and rejecting individual imperfect brick. Subject to the exceptions specified in Paragraph (*b*), individual brick may be culled and rejected for the following causes:

(*a*) *Variation in Size.*—Brick which vary from the nominal size specified in Section 5 by more than $\pm\frac{1}{8}$ in. in either transverse dimension, exclusive of kiln marks, or by more than $\pm\frac{1}{4}$ in. in length.

(*b*) *Broken or Chipped Brick.*—Brick which are chipped or broken so that neither wearing surface remains substantially intact, or so that the lower, or bearing surface is reduced in area more than 15 per cent, or are weakened structurally. However, brick so rejected but otherwise acceptable may be used for part-brick for beginning courses and for batting provided the portion so used meets all of the requirements for visual inspection.

(*c*) *Cracked Brick.*—Brick which are cracked to such a degree, either from shocks received in shipping and handling or from defective manufacture, as to produce defects described in Paragraph (*b*), unless such cracks are plainly superficial and insufficient to weaken the brick under the conditions of use.

(*d*) *Warped or Kiln-Marked Brick.*—Brick which are kiln-marked more than $\frac{3}{16}$ in. or which are misshapen, bent or twisted so that they will not form a proper surface, or align with other brick in conformance with the specifications for laying brick in the pavement. Vertical fiber brick shall have no kiln-marks in the wire cut surface.

(*e*) *Under-Burned Brick.*—Brick which are obviously too soft and too poorly vitrified to endure traffic wear. If disagreement on this requirement arises between the purchaser and the seller it shall be the right of the purchaser to make one or more rattler tests of the brick he wishes to exclude, and if in any or all tests the brick fail to conform to the requirements of these specifications, then all brick having the same objectionable appearance may be excluded.

ACCEPTANCE AND REJECTION

Basis of Acceptance

10. Acceptance of any lot of paving brick shall be based on the results of tests on samples representing that lot, subject only to rejection at any time of individually imperfect or unsatisfactory brick, in accordance with the requirements under Section 11.

Retesting

11. (*a*) If the percentage of loss in the rattler test on any sample exceeds by 2 per cent or less the maximum permissible rattler loss specified in Section 8 (*a*) for the size specified, or if the number of pieces weighing 1 lb. or more remaining after the completion of the rattler test exceeds 12 as specified in Section 8 (*b*), two additional tests shall be made. Each of these additional tests shall comply with the requirements of both Section 8 (*a*) and (*b*) and, in addition, the average rattler loss for the three tests shall not exceed the maximum permissible loss specified in Section 8 (*a*).

(*b*) Where a lot of paving brick is rejected for failure to meet the test requirements prescribed in these specifications, the purchaser may, at his option, permit the seller to regrade the rejected brick and again offer the brick for test and acceptance. The regraded brick shall be resampled and tested and if they again fail, the purchaser may definitely and finally reject the lot for which the test is representative.

Payment for Cost of Testing

12. Unless otherwise agreed, the seller shall furnish at the place of manufacture, without charge to the purchaser, the use of an approved rattler in efficient operating condition, conforming to the requirements specified in Section 15. Payment of the cost of testing shall be made by the purchaser, except that the cost of testing additional samples in accordance with Section 11, made necessary by the failure of any lot or lots, or of testing supplemental samples to determine the admissibility of a portion of any lot or lots, shall be paid by the seller; provided, however, that if the supplemental tests show the brick under examination to be acceptable, then the cost of such tests shall be paid by the purchaser.

SAMPLING

Standard Sample

13. The standard sample for the rattler test shall consist of ten whole undamaged brick which conform individually to the visual inspection requirements specified in Section 9.

Selection of Sample

14. (*a*) Samples shall preferably be selected at the place of manufacture and tested prior to shipment. However, in special cases where less than 100,000 brick are involved, samples, when required, may be selected subsequent to delivery at the destination.

Number of Standard Samples

(*b*) In general, one standard sample shall be selected for each 15,000 brick or fraction thereof; provided, however, that when the sampling is done at the place of manufacture prior to shipment and the total number of brick involved is more than 100,000 brick, the number of brick for which one standard sample is considered representative may be increased at the option of the purchaser to not more than 50,000 brick.

RATTLER TEST

Apparatus

15. The machine[1] used in the rattler test shall be of good mechanical construction, self-contained, and shall conform to the following details of material and dimensions, and shall consist of barrel, frame and driving mechanism as described in Sections 16 to 20.

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[1] A detailed illustration of a rattler which will meet the requirements specified appears in Plate I of the 1936 Book of A.S.T.M. Standards, Part II, p. 128. A copy of this plate showing the construction of the machine is available from the Headquarters of the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Barrel

16. The barrel of the machine shall consist of the heads, headliners, staves, and staveliners conforming to the following requirements:

(*a*) *Heads.*—The heads may be cast in one piece with the trunnions which shall be $2\frac{7}{16}$ in. in diameter, and shall have a bearing 6 in. in length; or they may be cast with heavy hubs, which shall be bored out for $2\frac{7}{16}$-in. shafts and shall be key-seated for two keys, each $\frac{1}{2}$ by $\frac{3}{8}$ in. and spaced 90 deg. apart. The shaft shall be a snug fit and when keyed shall be entirely free from lost motion. The distance from the end of the shaft or trunnion to the inside face of the head shall be $15\frac{3}{8}$ in. in the head for the driving end of the rattler and $11\frac{3}{8}$ in. for the other head, and the distance from the face of the hubs to the inside face of the heads shall be $5\frac{1}{8}$ in.

The heads shall not be less than $\frac{3}{4}$ nor more than $\frac{7}{8}$ in. in thickness. In outline, each head shall be a regular 14-sided polygon inscribed in a circle $28\frac{3}{8}$ in. in diameter. Each head shall be provided with flanges not less than $\frac{3}{4}$ in. in thickness and extending outward $2\frac{1}{2}$ in. from the inside face of the head to afford a means of fastening the staves. The surface of the flanges of the head shall be smooth and give a true and uniform bearing for the staves. To secure the desired true and uniform bearing, the surfaces of the flanges of the head shall be either ground or machined. The flanges shall be slotted on the outer edge, so as to provide for two $\frac{3}{4}$-in. bolts at each end of each stave, the slots to be $\frac{13}{16}$ in. in width and $2\frac{3}{4}$ in. center to center. Each slot shall be provided with a recess for the bolt head, which shall act to prevent the turning of the same. Between each two slots there shall be a brace $\frac{3}{8}$ in. in thickness extending down the outward side of the head not less than 2 in.

(*b*) *Headliners.*—There shall be for each head a cast-iron headliner, 1 in. in thickness and conforming to the outline of the head, but inscribed in a circle $28\frac{1}{8}$ in. in diameter. This headliner shall be fastened to the head by seven $\frac{5}{8}$-in. cap screws, through the head from the outside. Whenever these headliners become worn down $\frac{1}{2}$ in. below their initial surface level, at any point of their surface, they shall be replaced with new ones. The metal of these headliners shall be hard machinery iron and should contain not less than 1 per cent of combined carbon.

(*c*) *Staves.*—The staves shall be made of 6-in. medium-carbon steel structural channels $27\frac{1}{4}$ in. in length and weighing 15.5 lb. per lineal foot. The staves shall have two holes $\frac{13}{16}$ in. in diameter, drilled in each end, the center line of the holes being 1 in. from the end and $1\frac{3}{8}$ in. either way from the longitudinal center line. The spaces between the staves shall be as uniform as practicable, but shall not exceed $\frac{5}{16}$ in.

(*d*) *Staveliners.*—The interior or flat side of each stave shall be protected by a liner $\frac{3}{8}$ in. thick by $5\frac{1}{2}$ in. wide by $19\frac{3}{4}$ in. long. The liner shall consist of medium-carbon steel plate, and shall be riveted to the channel by three $\frac{1}{2}$-in. rivets, one of which shall be on the center line both ways and the other two on the longitudinal center line and spaced 7 in. from the center each way. The rivet holes shall be countersunk on the face of the liner and the rivets shall be driven hot and chipped off flush with the surface of the liners. These liners shall be inspected from time to time, and if found loose shall be at once re-riveted.

Preliminary Operation

17. When a new rattler, in which a complete set of new staves is furnished, is first put into operation, it shall be charged with 400 lb. of shot of the same sizes, and in the same proportion as provided in Section 21, and shall then be run for 18,000 revolutions at the rate of speed specified in Section 25 *(a)*. The shot shall then be removed and a standard shot charge inserted after which the rattler may be charged with brick for a test.

Relining

18. No stave shall be used for more than 70 consecutive tests without renewing its lining. Two of the 14 staves shall be removed and relined at a time in such a way that of each pair, one falls upon one side of the barrel and the other upon the opposite side, and also so that the staves changed shall be consecutive, but not contiguous: for example, 1 and 8, 3 and 10, 5 and 12, 7 and 14, 2 and 9, 4 and 11, 6 and 13, etc., to the end that the interior of the barrel at all times shall present the same relative conditions of repair. The changes in the staves should be made at the time when the shot charges are being corrected, and the record must show the number of charges run since the last pair of new lined staves was placed in position.

The staves when bolted to the heads shall form a barrel 20 in. in length, inside measurement, between headliners. The liners of the staves shall be so placed as to drop between the headliners. The staves shall be bolted tightly to the heads by four $\frac{3}{4}$-in. bolts and each bolt shall be provided with a locknut and shall be inspected at not less frequent intervals than every fifth test and all nuts kept tight. A record shall be made after each inspection showing in what condition the bolts were found.

Barrel Frame

19. The barrel shall be mounted on a cast-iron frame of sufficient strength and rigidity to support it without undue vibration. It shall rest on a rigid foundation with or without the interposition of wooden plates, and shall be fastened thereto by bolts at not less than four points.

Driving Mechanism

20. The barrel shall be driven by gearing whose ratio of driver to driven is not less than one to four. The counter shaft upon which the driving pinion is mounted shall not be less than $1\frac{15}{16}$ in. in diameter, with bearing not less than 6 in. in length. If a belt drive is used, the pulley shall be not

less than 18 in. in diameter and 6½ in. in face. A belt at least 6 in. in width, properly adjusted, to avoid unnecessary slipping, should be used.

Abrasive Charge

21. The abrasive charge shall consist of cast-iron spheres of two sizes.

(*a*) *Large Spheres.*—When new, the larger spheres shall be 3.75 in. in diameter and shall weigh approximately 7.5 lb. (3.40 kg.) each. Ten spheres of this size shall be used. These shall be weighed separately after each ten tests, and if the weight of any large sphere falls to 7 lb. (3.175 kg.) it shall be discarded and a new one substituted; provided, however, that all of the large spheres shall not be discarded and substituted by new ones at any single time, and that in so far as possible the large spheres shall compose a graduated series in various stages of wear.

(*b*) *Small Spheres.*—When new, the smaller spheres shall be 1.875 in. in diameter and shall weigh approximately 0.95 lb. (0.43 kg.) each. In general, the number of small spheres in a charge shall not fall below 245 nor exceed 260. The collective weight of the large and small spheres shall be as nearly 300 lb. as possible. No small sphere shall be retained in use after it has been worn down so that it will pass a circular hole 1.75 in. in diameter, drilled in an iron plate ¼ in. in thickness, or weigh less than 0.75 lb. (0.34 kg.).

Further, the small spheres shall be tested by passing them over the plate mentioned or by weighing, after every ten tests, and any which pass through or fall below the specified weight, shall be replaced by new spheres, provided, further, that all of the small spheres shall not be rejected and replaced by new ones at any one time, and that so far as possible the small spheres shall compose a graduated series in various stages of wear. At any time that any sphere is found to be broken or defective it shall at once be replaced.

Chemical Composition of Charge

22. (*a*) The cast-iron abrasive spheres shall conform to the following requirements as to chemical composition:

Combined carbon, min., per cent	2.50
Graphitic carbon, max., per cent	0.25
Silicon, max., per cent	1.00
Manganese, max., per cent	0.50
Phosphorus, max., per cent	0.25
Sulfur, max., per cent	0.08

(*b*) For each new batch of spheres used, the chemical analysis shall be furnished by the maker or be obtained by the user, before introducing the spheres into the charge, and unless the analysis conforms to the requirements specified, the batch of spheres shall be rejected.

Scales

23. The scales used shall be placed on a solid level foundation and shall be sufficiently sensitive to determine the initial weight of 10 bricks to the nearest 0.1 lb. The scale shall be tested by a standard test weight at intervals of not more than every ten tests.

Test Specimen

24. The number of brick per test shall be ten whole bricks for all weights of brick, and the brick shall be weighed to the nearest 0.1 lb. No brick should be selected as part of a regular test that would be rejected by any other requirements of the specifications under which the purchase is made.

Procedure

25. *(a)* The test shall consist of 1800 revolutions of the rattler at the rate of not less than 29.5 nor more than 30.5 r.p.m. A counting machine shall be attached to the rattler for counting the revolutions. A margin not to exceed ten revolutions will be allowed for starting and stopping. Only one start and stop per test is, in general, acceptable. If, from accidental causes, the rattler is stopped and started more than once during a test, and the percentage loss exceeds the maximum permissible under the specifications, the test shall be disqualified and another made.

(b) Any test, at the expiration of which a staveliner is found detached from the stave or seriously out of position, shall be rejected.

Percentage Loss of Weight

26. The loss shall be calculated as a percentage of the original weight of the brick composing the charge, by weighing the brick remaining in the rattler at the end of the test, deducting this weight from the original weight of the brick, dividing this difference by the original weight of the brick and multiplying by 100, as follows:

$$\text{Percentage of Loss} = \frac{\text{Original Weight} - \text{Weight after Testing}}{\text{Original Weight}} \times 100$$

In weighing the rattled brick, any piece weighing less than 1 lb. shall be rejected.

Rattler Record

27. A complete and continuous record shall be kept of the operation of all rattlers working under the rattler test. This record shall contain the following data concerning each test made:

1. The name of the person, firm or corporation furnishing each sample tested.
2. The name of the maker of the brick represented in each sample tested.
3. The name of the street or contract, which the sample represented.
4. The brands or marks upon the bricks by which they were identified.
5. The number of bricks furnished.
6. The date on which they were received for test.

7. The date on which they were tested.
8. The drying treatment given before testing, if any.
9. The length, width and thickness of the bricks.
10. The collective weight of the ten large spherical shot used in making the test at the time of their last standardization.
11. The number and collective weight of the small spherical shot used in making the test at the time of their last standardization.
12. The total weight of the shot charge, after its last standardization.
13. Certificate of the operator that he examined the condition of the machine as to staves, liners, and any other parts affecting the barrel, and found them to conform to the requirements at the beginning of the test.
14. Certificate of the operator of the number of charges tested since the last standardization of shot charge and last renewals of stave liners.
15. The time of the beginning and ending of each test, and the number of revolutions made by the barrel during the test, as shown by the counting machine.
16. Certificate of the operator as to number of stops and starts made in each test.
17. The initial collective weight of the ten brick composing the charge and their collective weight after rattling.
18. The number of broken bricks and remarks upon the portions which were included in the final weighing.
19. General remarks upon the test and any irregularities occurring in its execution.
20. The certificate of the operator that the test was made in accordance with the requirements of the American Society for Testing Materials and that the record is a true record.
21. The signature of the operator or person responsible for the test.
22. The location of the laboratory where the test was made and name of the owner.
23. The serial number of the test.

In the event of more than one copy of the record of any test being required, they may be furnished on separate sheets and marked duplicate, but the original record shall always be preserved intact and complete.

TENTATIVE SPECIFICATIONS
FOR
SEWER BRICK (MADE FROM CLAY OR SHALE)[1]

A.S.T.M. Designation: C 32 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1921; ADOPTED, 1923; REVISED, 1924;
REISSUED AS TENTATIVE, 1937.

Scope

1. These specifications cover brick made from clay or shale and burned, to be used in drainage structures for the conveyance of sewage, industrial wastes, and storm water. Three grades of brick are covered as follows:

Grade SA.—Brick intended for use in structures requiring imperviousness and resistance to the action of sewage carrying large quantities of abrasive material at velocities exceeding 8 ft. per sec.

Grade MA.—Brick intended for use in structures requiring imperviousness and resistance to the action: (*a*) of sewage free from abrasive materials, and (*b*) of sewage carrying abrasive materials at velocities of 8 ft. per sec. or less.

Grade NA.—Brick intended for use in structures not requiring high degrees of imperviousness nor of abrasive resistance.

NOTE.—Bricks of Grade *NA* are suitable for use in catch basins, arches, the upper portions of manholes, and for backing.

Properties

2. (*a*) The brick shall conform to the physical requirements for the grades specified, as follows:

	Compressive Strength, lb. per sq. in., average gross area		Water Absorption by 5-hr. boiling, per cent	
	Average of 5 Bricks	Individual Minimum	Average of 5 Bricks	Individual Maximum
Grade *SA*	8000	5000	6	9
Grade *MA*[a]	5000	3000	12	16
Grade *NA*[a]	2500	2000	20	24

[a] Where resistance to frost action in the presence of moisture is required, Grades *MA* and *NA* shall conform to the additional requirement that the ratio of absorption by 24-hr. submergence in cold water to that after 5 hr. in boiling water, *C*/*B* ratio, shall not exceed 0.80.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

These specifications are in effect a revision of and replace the former Standard Specifications for Clay Sewer Brick (C 32 - 24), which standard was accordingly discontinued in 1937.

(*b*) Unless otherwise specified by the purchaser, bricks of Grade *SA* shall be accepted in lieu of Grade *MA;* also Grades *SA* and *MA* shall be accepted in lieu of Grade *NA*.

Test Specimens

3. (*a*) For purpose of tests, bricks representative of the commercial product shall be selected by a competent person appointed by the purchaser, the place or places of selection to be designated when the purchase order is placed. The manufacturer or seller shall furnish specimens for tests without charge. All bricks shall be carefully examined and their condition noted before testing.

(*b*) For the purpose of tests, not less than five brick shall be required for each investigation. In general, two samples of five brick each shall be tested for each 100,000 bricks contained in the lot under consideration; but where the total quantity exceeds 500,000, one sample of five brick from each 100,000 shall be tested. Additional representative samples may be taken at any time or place at the discretion of the purchaser.

Sizes

4. Brick of one or more of the following designated sizes as specified shall be furnished:

Size	Depth, in.	Width, in.	Length, in.
No. 1	$2\frac{1}{4}$	$3\frac{3}{4}$	8
No. 2	$2\frac{1}{2}$	4	$8\frac{1}{2}$
No. 3	3	4	$8\frac{1}{2}$
No. 4	$3\frac{1}{2}$	4	$8\frac{1}{2}$

Permissible Variations in Size

5. For any lot of sewer brick furnished under these specifications, not more than 2 per cent of the bricks shall vary from the nominal size requirements specified in Section 4 by more than $\pm \frac{1}{8}$ in. in either transverse dimension, or by more than $\pm \frac{1}{4}$ in. in length.

Materials and Workmanship

6. (*a*) The brick shall be manufactured from surface clay, fire clay, or shale, or a combination of these materials. The composition shall be such that the finished brick are free from deleterious materials including coarse grains, stones, pebbles, or visible grains of lime.

(*b*) Brick shall be of rectangular cross-section with substantially straight, square corners. The ends and at least one edge shall have plain surfaces.

(*c*) When paving brick are used, lugs and grooves will be permitted on one edge and the ends. Kiln marks not exceeding $\frac{3}{16}$ in. in depth shall be permitted on the opposite edge.

(*d*) Brick shall be free from checks or cracks extending into the body of the brick in a manner that would affect their serviceability or strength.

NOTE.—Where cored brick are permitted by the purchaser, they shall conform to the requirements of these specifications.

Methods of Testing

7. Brick shall be tested in accordance with the Standard Methods of Testing Brick (Modulus of Rupture, Compressive Strength, Absorption) (A.S.T.M. Designation: C 67) of the American Society for Testing Materials.[1]

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[1] 1937 Supplement to Book of A.S.T.M. Standards, p. 78.

TENTATIVE SPECIFICATIONS AND METHODS OF TEST
FOR
CONCRETE UNITS FOR NON-LOAD-BEARING MASONRY[1]

A.S.T.M. Designation: C 129 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

SPECIFICATIONS

Scope

1. These specifications apply to strength, moisture content and general requirements of hollow non-load-bearing concrete masonry units made from portland cement and suitable aggregates such as sand, gravel, crushed stone, bituminous or anthracite cinders, burned clay or shale, and blast-furnace slag.

Compressive Strength

2. The average compressive strength of five units at the time of delivery to the site shall not be less than the following:

	MEAN OF FIVE TESTS	INDIVIDUAL MINIMUM
Compressive strength, based on gross area, lb. per sq. in.	350 or over	300

Moisture Content

3. The average moisture content in the units at time of sampling shall not exceed 40 per cent of the total absorption of the units when calculated as described in Section 25.

NOTE.—It is recommended that units be stored under cover or otherwise protected against wetting until laid in the wall.

Dimensions and Permissible Variations

4. (*a*) No over-all dimension shall vary more than 3 per cent over or under the specified dimension for any form or size of unit.

(*b*) Minimum face shell thickness shall be not less than ½ in. at any point.

Materials, Workmanship and Finish

5. (*a*) Units shall contain no materials which will stain plaster applied thereto. The combustible content present in cinder aggregate shall not exceed 35 per cent of the weight of the aggregate.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

(*b*) All units shall be sound and free from cracks or defects that would interfere with the proper placing of the unit.

(*c*) Units which are intended to serve as a base for plaster or stucco shall have a sufficiently rough surface to give good bond.

(*d*) If a particular surface texture, finish, or uniformity of color is required, this shall be as specified in the purchase order.

Marking

6. All units shall bear a distinctive mark of the manufacturer or shall otherwise be readily identified as to origin.

Inspection

7. The purchaser or his authorized representative shall be afforded proper facilities to inspect the units both at the factory and at the site of the work.

Rejection

8. Individual units shall be rejected for failure to meet materials, size, or workmanship and finish requirements. In case of failure to meet the requirements for strength or moisture content, or both, the manufacturer may sort the shipment and new samples shall be selected by the purchaser from the retained lot and tested at the expense of the manufacturer. In case the second set of samples fails to meet the test requirements, the entire lot shall be rejected.

Expense of Tests

9. Except as specified in Section 8 and unless otherwise agreed, the expense of inspection and testing shall be borne by the purchaser.

METHODS OF TESTING

SAMPLING

Selection of Samples for Test

10. Samples of full-size units for testing shall be selected by the purchaser or by his authorized representative at the factory or at the site of the work, as specified. If at the site, units for moisture content tests shall be selected within 48 hr. after delivery. Samples shall be representative of the whole lot of units from which they are selected.

Number of Samples

11. For the strength and moisture content determinations, ten individual units shall be selected from each lot of 10,000 units or fraction thereof; 20 individual units from each lot of more than 10,000 and less than 100,000 units. For lots of more than 100,000 units, there shall be selected 10 individual units from each 50,000 units contained in the lot.

Marking Samples

12. All units selected for purpose of tests shall be permanently identified and accurately weighed before shipment to the laboratory.

Time Allowed for Testing

13. At least 10 days shall be allowed for the completion of the tests.

COMPRESSIVE STRENGTH TESTS

Test Specimens

14. Five full-size units, selected in accordance with Sections 10 to 13, shall be tested.

Position of Specimens

15. All specimens shall be tested in a position such that the load is applied to the unit in the same direction as in service.

Capping Test Specimens

16. (*a*) Bearing surfaces of the test specimens and portions adjoining them, which are liable to absorb water from the plastic capping, shall be coated with shellac and allowed to dry.

(*b*) A quantity of plastic mortar made of a mixture of one part (by volume) of portland cement and one part of unretarded gypsum (plaster of Paris) mixed with sufficient water to spread evenly shall be placed on a plane surface which has been coated with oil. The surface to be capped shall be placed on this mortar, and while holding the specimen so that its axis is at right angles to the capping surface it shall be firmly pressed down with a single motion.

(*c*) The average thickness of the cap after the extruded plaster has been removed and the edges trued shall not be more than $\frac{1}{8}$ in. Patching of caps after setting shall not be permitted. Imperfect caps shall be removed and replaced with new ones.

(*d*) *Time of Testing.*—The cap shall be allowed to age at least 6 hr. before the specimen is tested.

Bearing Block

17. A spherical bearing block of proper design shall be placed on top of the test specimen. When the bearing area of the spherical bearing block is not sufficient to cover the area of the specimen, metal plates of sufficient thickness to prevent appreciable bending shall be placed between the spherical bearing block and the specimen.

Speed of Testing Machine

18. The speed of the moving head of the testing machine shall not be more than 0.05 in. per min.

Calculation

19. The compressive strength of a specimen shall be the maximum applied load in pounds divided by the gross cross-sectional area of the unit in square inches. The gross area of a unit is the total area of a section perpendicular to the direction of the load, including areas within cells and within re-entrant spaces unless these spaces are to be occupied in the masonry by portions of the adjacent masonry.

MOISTURE CONTENT

Test Specimens

20. Five full-size units, selected in accordance with Sections 10 to 13, shall be tested.

Marking Test Specimens

21. Each test specimen shall be marked so that it may be identified at any time. Markings shall not cover more than 5 per cent of the total superficial area of the specimen.

Drying Test Specimens

22. Preparatory to the moisture content tests, all specimens shall be dried in a drier or oven at a temperature between 100 and 115 C. (212 and 240 F.) and weighed at intervals until the loss in weight does not exceed 1 per cent in a 24-hr. period.

Accuracy of Weighings

23. The balance used shall be accurate to within 0.5 per cent of the weight of the specimen.

Saturation of Specimens

24. The dry specimens shall be completely immersed in water at a temperature between 15.6 and 26.7 C. (60 and 80 F.) for 24 hr. They shall be removed from the water and allowed to drain for 1 min. by placing on a $\frac{3}{8}$-in. or coarser wire mesh, visible surface water being removed with a damp cloth. The specimens shall then be immediately weighed.

Calculation

25. The moisture content of the units at time of sampling (in per cent of the total absorption) is the difference between the average weight when sampled (Section 12) and the average dry weight (Section 22) divided by the difference between the average wet weight (Section 24) and the average dry weight and multiplied by 100, as follows:

$$\text{Percentage of moisture} = \left(\frac{\text{Sampled weight} - \text{Dry weight}}{\text{Wet weight} - \text{Dry weight}}\right) \times 100$$

TENTATIVE SPECIFICATIONS

FOR

FIRE-RETARDANT PROPERTIES OF WOOD FOR SCAFFOLDING AND SHORING[1]

A.S.T.M. Designation: C 132 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications apply to the properties of wood scaffolding and shoring processed with fire-retardant chemicals so as to protect the wood from the usual fire hazards incident to construction operations.

Processing

2. The processing shall not affect the strength of the wood or otherwise render it unsuitable for the purposes for which it is intended as determined by the authorities having jurisdiction.

Fire-Retardant Properties

3. The processed wood, when subjected to the timber test described in Sections 8 and 9, shall meet the following requirements as to fire-retardant properties:

(*a*) *Duration of Flame.*—The average continuation of flaming (Note 1) shall not exceed 30 sec.

(*b*) *Duration of Glow.*—The average continuation of glow (Note 2) shall not exceed an additional 30 sec. after the cessation of flaming.

(*c*) *Loss in Weight.*—The average loss in weight (Note 3) shall not exceed 30 per cent.

NOTE 1: *Duration of Flame.*—The continuation of flaming by the material after removal from the igniting flame indicates the degree of combustibility, the ignition resistance and the tendency of the material to spread fire.

NOTE 2: *Duration of Glow.*—The tendency of the material to glow after flaming ceases is indicative of the presence of combustible material and the possibility of fire spread through smoldering.

NOTE 3: *Loss in Weight.*—The loss in weight is a measure of the combustion loss and indicates the loss in strength or structural qualities and the contribution of fuel to the fire.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-5 on Fire Tests of Materials and Construction.

Marking

4. (*a*) The wood shall be branded with the name of the manufacturer and the year and month of processing.

(*b*) The material shall be retested at intervals of one year and if it passes the tests prescribed in these specifications it shall be rebranded with the latest date of test.

Sampling

5. Samples for test shall be selected by and tested under the supervision of the authority having jurisdiction. A minimum of two samples shall be selected from each lot or two samples for each 5000 board feet, or fraction thereof, where the quantity exceeds this amount.

Test Specimens

6. Specimens for the timber test shall be $\frac{3}{4}$ in. in thickness, $1\frac{1}{2}$ in. in width and 10 in. in length. Each specimen shall be taken from the edge of and not nearer than 2 ft. from the end of each sample selected for test and shall be carefully cut from the narrow face of the sample.

Conditioning of Test Specimens

7. (*a*) Test specimens shall be dried at a temperature of 150 F. (66 C.) to a moisture content of 7 per cent ±1 per cent, as determined on a representative moisture specimen tested in accordance with Paragraph (*b*). The representative moisture specimen for the purpose of determining moisture content shall consist of a full-size specimen selected in accordance with Section 6.

(*b*) All loose splinters shall be removed immediately from each moisture specimen and the weight determined. The moisture specimens shall be open piled in a ventilated oven and dried at a temperature which shall ultimately be increased to 208 F. ±6 F. (98 C. ±3 C.) until constant weight is obtained, after which the resulting weight shall be carefully determined and recorded as the oven-dry weight. For specimens not over 1 in. in length (in the direction of grain) about 48-hr. continuous drying under the conditions specified is sufficient to bring the specimens to constant weight. For drying longer specimens, considerably more time will be required, since the rate of drying from the side grain surface of wood is only about one-sixth that from the end surface.

(*c*) The loss in weight expressed as a percentage of the oven-dry weight as determined in Paragraph (*b*) shall be considered the moisture content of the specimen.

Apparatus for Timber Test

8. (*a*) *Gas Crucible Furnace.*—A 6-in. gas crucible furnace will be required. A furnace that has been found satisfactory is shown in Fig. 1. The furnace proper consists of a bottom refractory cylinder with burner

tips projecting into it and an upper refractory cylinder 6 in. in height and 6 in. in inside diameter which rests upon the bottom cylinder. The burner is held in position under the table and three burner tips project through slots tightly fitted with fire clay so as to make a tight joint between the tips and the lining. The tips direct the flame so as not to strike the crucible but to rotate around it.

(*b*) *Fumehood.*—The fumehood in which the test is conducted shall be equipped with a device for controlling the ventilation. The velocity of the ventilating air shall not exceed 20 lineal feet per minute at the center of the hood.

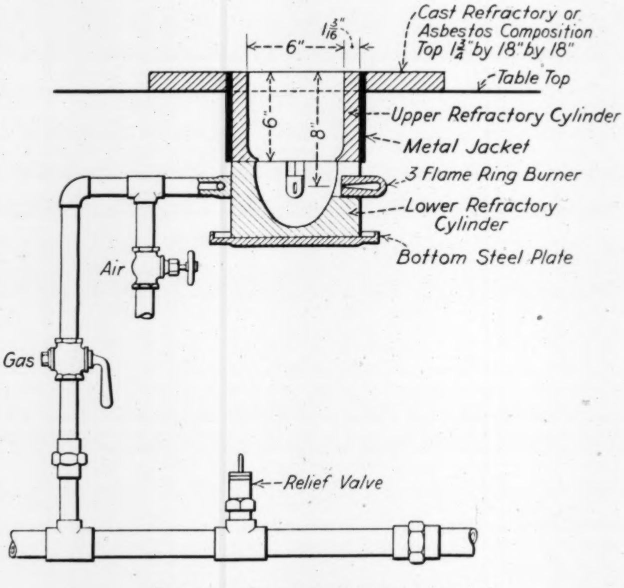
$1\frac{3}{16}$"
6"
Cast Refractory or Asbestos Composition Top $1\frac{3}{4}$" by 18" by 18"
Table Top
6"
8"
Upper Refractory Cylinder
Metal Jacket
3 Flame Ring Burner
Lower Refractory Cylinder
Bottom Steel Plate
Air
Gas
Relief Valve

FIG. 1.—Gas Crucible Furnace.

(*c*) *Miscellaneous Apparatus.*—The balance used in the measurement of weight shall be accurate within 0.1 g. Temperature measurements should be obtained by means of a potentiometer or other approved means of securing temperature readings.

Timber Test

9. Two weighed test specimens shall be placed side by side across the top of a 6-in. gas crucible furnace with the uncut faces down and their narrow uncut edges together. The specimens shall be flanked by fire brick on each side and at one end. In this position they shall be exposed for 2 min. to a furnace temperature of 1700 F. ± 25 F. (925 C. ± 14 C.) as measured by an 8-gage thermocouple terminating at the center of the furnace opening, 1 in. below the line of contact of the test specimens.

The specimens shall then be removed from the furnace and placed upon a fire brick alongside the furnace in the fumehood with the burned faces up and the time during which flaming and glow continues on each specimen shall be recorded. The test specimens shall then be reweighed and the percentage loss in weight of each specimen shall be calculated. The results of the tests for duration of flame, duration of glow, and loss in weight of the two test specimens shall each be averaged and recorded.

Inspection and Rejection

10. (*a*) Tests shall be made on samples taken at the processing plant before shipment of the processed material.

(*b*) Check tests shall be permissible on samples taken at the place of use.

(*c*) Material failing to meet the requirements of these specifications shall be rejected, and shall be kept from or removed from the place of use.

TENTATIVE METHODS OF CHEMICAL ANALYSIS
OF
REFRACTORY MATERIALS[1]

A.S.T.M. Designation: C 18 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1917; ADOPTED IN AMENDED FORM, 1920; REVISED, 1921, 1935; REVISED AND REISSUED AS TENTATIVE, 1937.

Scope

1. (*a*) These methods describe procedures for the chemical analysis of fireclay, silica, and high-alumina refractories; chrome ores and chrome brick, and magnesite refractories.

(*b*) The analytical procedures appear in the following order:

Fireclay Refractories: SECTION
- Moisture 7
- Loss on Ignition 8
- Silica 9
- Iron, Aluminum, and Titanium Oxides 10
- Iron Oxide 11
- Titania 12
- Alumina 13
- Lime 14
- Magnesia 15
- Alkalies 16

Silica Refractories:
- Moisture 18
- Loss on Ignition 19
- Silica 20
- Iron and Aluminum Oxides 21
- Iron Oxide 22
- Titania 23
- Alumina 24
- Lime 25
- Magnesia 26
- Alkalies 27

High-Alumina Refractories:
- Moisture 29
- Loss on Ignition 30
- Silica 31
- Separation of Al_2O_3 and P_2O_5 from Fe_2O_3, TiO_2, CaO, and MgO 32
- Iron Oxide 33
- Titania 34
- Phosphorus Oxide 35
- Alumina 36
- Lime 37
- Magnesia 38
- Alkalies 39

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

Chrome Ores and Chrome Brick:

SECTION
A. Procedure for Complete Analysis 40
B. Rapid Routine Method 42
Chromium Oxide 43
Silica and Iron Oxide 44

Magnesite Refractories:

Moisture 47
Loss on Ignition 48
Silica 49
Iron and Aluminum Oxides 50
Iron Oxides 51
Alumina 52
Lime 53
Magnesia 54
Manganese as MnO 55

PREFACE

The following methods have been compiled as standard procedures for use in referee analyses. These methods, however, where the determination of iron oxide as Fe_2O_3 is involved, are not intended to preclude the use of other methods that give results within the permissible tolerances. For the sake of uniformity the classical Zimmerman-Reinhart procedure is specified for the determination of iron oxide. It is recognized that numerous other procedures are equally accurate and often more convenient. The other procedures commonly in use include reduction of an oxidized solution with zinc, or other metal, and titration with standard $KMnO_4$, or titration with a standard solution of titanous chloride in an oxidized solution. These procedures shall be considered acceptable provided the analyst has obtained results by his special procedure which check with the Zimmerman-Reinhart procedure within the limits specified in Section 4. It is suggested that the National Bureau of Standards' standard samples be used for checking the accuracy of procedures.

It will be understood that the making of a complete analysis of a refractory material is a difficult procedure requiring a wide knowledge of the chemistry involved in the operations, and a thorough training in carrying out the work. A skilled analyst of good training is therefore required to do the work. The descriptions here given cover the vital points of procedure, but frequent reference in regard to the details of the various manipulations should be made to "Applied Inorganic Analysis" by Hillebrand and Lundell,[1] and to similar publications. Particularly in the determination of alumina, reference should be made to Scientific Paper No. 286 of the National Bureau of Standards.[2]

Preparation of Samples for Analysis

2. The sample, carefully secured after the manner of one of the approved methods of sampling, shall be crushed in a small jaw or roll-type crusher with hardened tool-steel faces to pass a No. 8 sieve.[3] The sample shall then be crushed to pass a No. 20 sieve,[3] mixed and quartered to about 50 g. This 50-g. sample shall then be ground so that it will all pass a No. 100 sieve,[3] unless otherwise specified, mixed thoroughly and placed in a container which will insure freedom from contamination. Fine grinding

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[1] W. F. Hillebrand and G. E. F. Lundell, "Applied Inorganic Analysis," Wiley and Son, New York City (1929).

[2] W. Blum, "Determination of Alumina as Oxide," National Bureau of Standards *Scientific Paper No. 286.*

[3] For detailed specifications for these sieves, see the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials, 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

shall be done in an agate mortar, either by hand or by a mechanical sample grinder of the McKenna, Carling, Fisher or similar type, so constructed as to prevent the introduction of impurity.

Statement of Analysis

3. Moisture shall be determined on the sample in its ordinary air-dried condition, and all other percentage compositions shall be calculated to a moisture-free basis. Whenever a sample is weighed out for any determination other than hygroscopic moisture, it shall be moisture-free. If preferred, the sample may be dried in a weighing bottle from which the required samples shall be weighed out.

Checking Results

4. In all cases, check determinations shall be made, and the results shall be redetermined if satisfactory checks are not obtained. It shall be considered satisfactory if the differences between check determinations do not exceed the following limits:

	PERMISSIBLE VARIATIONS BETWEEN CHECK DETERMINATIONS, MAX., PER CENT
For silica or other constituent amounting to 30 per cent[a] or over	0.3
For alumina or other constituent amounting to 10 to 30 per cent[a]	0.2
For any other constituent amounting to under 10 per cent[a]	0.1

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[a] These figures are stated in terms of the whole sample as 100 per cent.

Blank

5. Blank determinations on the reagents shall be made for each constituent in the refractory and this blank deducted in each case. For the determination of the silica blank, approximately 0.25 g. of Al_2O_3 should be added as pure aluminum chloride.

Reagents and Special Solutions Required

6. The lists in Paragraphs (*a*) to (*n*) include those reagents and special solutions common to two or more of the analytical procedures. Special reagents and solutions required will be found listed with the particular method in which they are prescribed.

(*a*) *Concentrated Acid and Ammonia Solutions.*—Concentrated c. p. acid and ammonia solutions of approximately the following specific gravities, or concentrations, will be required:

Hydrochloric acid, HCl	1.19 sp. gr.
Sulfuric acid, H_2SO_4	1.84 sp. gr.
Nitric acid, HNO_3	1.42 sp. gr.
Hydrofluoric acid, HF	40 per cent
Perchloric acid, $HClO_4$	60 to 70 per cent, c. p.[a]
Ammonium hydroxide, NH_4OH	0.90 sp. gr.
Sulfurous acid	6 per cent solution[b]

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[a] Lower purity varieties may contain aluminum oxide, Al_2O_3, as an impurity.

[b] As supplied by reagent manufacturers.

(*b*) *Diluted Acid and Ammonia Solutions.*—The diluted acid and ammonia solutions referred to are of varying percentages by volume. They shall be made up by mixing proportional volumes of the concentrated reagent and water. The diluted sulfuric acid mixtures shall be made up by slowly stirring the acid into the water. These diluted solutions are designated in the methods as (1:4), (1:9), etc., except very diluted solutions which are referred to by the percentage of reagent added. The designations in parenthesis indicates the ratio of the volume of the concentrated reagent to the volume of water; for example, H_2SO_4 (1:9) solution contains 10 per cent by volume of H_2SO_4 (sp. gr. 1.84). The following will be required:

	PERCENTAGE BY VOLUME
Hydrochloric acid	50 20
Sulfuric acid	50 10 5
Ammonium hydroxide	50 5

(*c*) *Ammonium Chloride Solution (2 per cent).*—Dissolve 2 g. of NH_4Cl in 100 ml. of water.

(*d*) *Ammonium Oxalate Solution (Saturated).*—Dissolve 4 g. of $(NH_4)_2C_2O_4$ in 100 ml. of water.

(*e*) *Standard Potassium Permanganate Solution (0.1 N).*—Dissolve 3.25 g. of $KMnO_4$ in 1000 ml. of water. Standardize against the National Bureau of Standards' standard sample 40*c* of sodium oxalate.

(*f*) *Standard Potassium Permanganate Solution (0.04 N).*—Dissolve 2.5 g. of $KMnO_4$ in water and make up to 2 liters. Allow to stand for a week, filter through an asbestos mat and keep in a dark place. Standardize against the National Bureau of Standards' standard sample 40*c* of sodium oxalate.

(*g*) *Standard Titania Solution.*—Weigh out 0.05 g. of pure calcined TiO_2. Fuse with 10 g. of $K_2S_2O_7$ in a clean platinum crucible, keeping the temperature as low as possible to maintain fluidity. Cool, and dissolve in about 300 ml. of a diluted H_2SO_4 solution containing 50 ml. of the concentrated acid. Cool, transfer to a 500-ml. volumetric flask, dilute to the mark with water and mix thoroughly. To standardize the solution, take two 50-ml. portions in 400-ml. beakers, dilute, boil, and precipitate with NH_4OH. Filter, and wash with hot water. Place the papers in the original beakers, add 15 ml. of HCl, stir to macerate the paper, dilute, and precipitate again with NH_4OH. Filter, and wash with hot water until free of alkali salts. Ignite carefully, blast, and weigh. From the weight determined, calculate the strength of the solution.

(*h*) *Stannous Chloride Solution.*—Dissolve 50 g. of $SnCl_2$ in 100 ml.

of HCl and dilute to 1000 ml. Keep a few pieces of metallic tin in the bottle.

(*i*) *Mercuric Chloride Solution.*—Prepare a saturated solution of $HgCl_2$.

(*j*) *Manganese Sulfate Solution.*—Dissolve 70 g. of crystalline $MnSO_4$ in 500 ml. of water. Add 140 ml. of phosphoric acid, H_3PO_4 (sp. gr. 1.7) to 130 ml. of H_2SO_4 (sp. gr. 1.84) and dilute to 1000 ml.

(*k*) *Hydrogen Peroxide Solution (30 per cent).*

(*l*) *Diammonium Phosphate Solution.*—Dissolve 10 g. of $(NH_4)_2HPO_4$ in 100 ml. of water.

(*m*) *Chloroplatinic Acid Solution.*—The solution commonly employed contains 1 g. of platinum in 10 ml. of solution.

(*n*) *Ethyl Alcohol Solution (80 per cent).*—Prepare a solution containing 80 per cent by volume of ethyl alcohol in water.

FIRECLAY REFRACTORIES

Moisture

7. Weigh 1.00 g. of the sample and heat at a temperature not under 105 nor over 110 C., to constant weight.

Loss on Ignition

8. Remove hygroscopic moisture by heating the air-dried sample for 2 hr. at 110 to 120 C. Then weigh 1.000 g. of the dried sample and heat to constant weight over a blast lamp, or in an electric furnace at 1000 C. Record the loss in weight as the ignition loss.

Silica

9. Weigh 0.5000 g. of the dry sample into a platinum crucible containing about 5 g. of powdered anhydrous Na_2CO_3 and mix well with a platinum wire. Cover the mixture with a little more Na_2CO_3. Heat gradually to the full heat of a good burner (1000 to 1100 C.) maintained for about 1 hr. until complete solution is obtained. Place the crucible cover on a triangle and when the melt has partially cooled pour it on the lid (Note). When cool, place the crucible and lid in a 150-ml. beaker, placing the button on a watch glass above the beaker. Add 30 ml. of HCl (1:1). When solution is complete wash off the crucible and lid with HCl (1:4), taking care to remove all silica. Place the button in the solution. Transfer the contents of the beaker to a porcelain evaporating dish and evaporate to dryness on a steam bath. Bake for 1 hr. at 110 C. Add 20 to 30 ml. of HCl (1:1) and 50 ml. of hot water. When all salts have been dissolved allow to settle for several minutes and then filter through a No. 40 Whatman paper or equivalent. Wash the silica three times by decantation using 20 to 30-ml. portions of first hot water, then HCl (1:1) and again with hot water. Transfer the precipitate to

the filter paper, removing all silica from the dish with a policeman. Wash the paper and precipitate with hot water until free from salt. To recover the small amount of silica remaining in the filtrate, evaporate to dryness, using the same procedure for baking and filtering as before. Combine the two precipitates, place in a platinum crucible and burn off the paper carefully to prevent any loss of silica. Ignite the sample at 1100 to 1200 C. to constant weight (15 to 20 min. is usually sufficient), cool in a desiccator, and weigh. Moisten the residue with several milliliters of water, add 10 ml. of HF and 3 or 4 drops of H_2SO_4. Evaporate the solution to dryness, ignite carefully to prevent decrepitation and blast for several minutes at 1100 C. Cool the crucible in a desiccator, weigh, and repeat blasting to constant weight. The loss in weight from the original silica residue represents the silica content, except for that part of the silica which is later recovered from alumina, etc.

NOTE.—Another scheme to aid in subsequent solution of the fused melt is to rotate the crucible as it cools spreading the mass up the side walls.

Iron, Aluminum, and Titanium Oxides

10. Fuse the residue with 1 g. of fused potassium bisulfate ($K_2S_2O_7$), dissolve in a small amount of water and add to the filtrate from the silica determination. Add 5 g. of NH_4Cl and 3 drops of methyl red solution (0.1 per cent). Heat the solution almost to boiling and add slowly NH_4OH (1:1) until the indicator has changed to a yellow color. Boil for several minutes to remove the excess ammonia. Allow to settle for 30 min. and decant through a No. 41 Whatman filter paper or equivalent, transferring the precipitate to the paper and washing the beaker and paper several times with a warm NH_4Cl solution (2 per cent). Reserve the filtrate for the determination of CaO and MgO (Section 14). Return the precipitate to the original beaker, add 50 ml. of hot water, and 10 ml. of HCl (sp. gr. 1.19). Stir until the precipitate is dissolved and the paper is well macerated. Dilute to about 200 ml. with hot water; precipitate and filter as before. Wash the paper and precipitate with a warm NH_4Cl solution (2 per cent). Place the precipitate in a weighed platinum crucible, and ignite. Continue the ignition at 1200 C. to constant weight (15 to 20 min. is usually sufficient). Cool in a desiccator, and weigh with the crucible covered with the lid. The R_2O_3 consists of the Al_2O_3, TiO_2, and Fe_2O_3 present in the sample. In addition there may be small amounts of P_2O_5, ZrO_2, V_2O_5, and Cr_2O_3.

Iron Oxide

11. (*a*) *Method A. Fe_2O_3 Determined on R_2O_3 Sample.*—Heat the R_2O_3 precipitate (Note) obtained in the determination of iron, aluminum and titanium oxides (Section 10) with fused $K_2S_2O_7$ (moisture free) until solution is complete. Dissolve the fusion in 50 ml. of H_2SO_4 (1:9) and evaporate to fumes. Cool, dilute with water and filter off the silica, wash-

ing with hot water. Reserve the filtrate for the determination of Fe_2O_3 and TiO_2. Ignite the silica in a platinum crucible and weigh. Treat the precipitate with 5 ml. of HF and 2 or 3 drops of H_2SO_4. Evaporate to dryness, ignite, and weigh. The loss in weight represents extra silica which should be added to that determined previously and also deducted from the weight of the R_2O_3 precipitate. Evaporate the filtrate obtained in correcting the R_2O_3 precipitate for silica to about 75 ml. Cool, and dilute to 100 ml. in a volumetric flask. Reserve 25 ml. for the determination of TiO_2 (Section 12). To the remainder, add 25 ml. of HCl (1:1) and heat to boiling. Reduce the iron by adding $SnCl_2$ solution, drop-by-drop from a pipette with constant swirling of the beaker, until the solution is colorless. Then add one drop in excess. Cool quickly in running water, then add at one stroke 15 ml. of saturated $HgCl_2$ solution. Allow to stand for 3 min., transfer with washing to a 1000-ml. beaker containing 300 ml. of cold distilled water, and 25 ml. of $MnSO_4$ solution. Titrate with standard 0.04 *N* $KMnO_4$ solution added very slowly while stirring until a permanent pink endpoint is obtained.

NOTE.—Instead of fusing directly in the platinum crucible in which the R_2O_3 was ignited, the precipitate may be brushed into a porcelain crucible and then fused with $K_2S_2O_7$. This avoids loss of platinum by the action of the bisulfate, and no platinum is present in the filtrate to interfere with the iron determination.

(*b*) *Method B. Fe_2O_3 Determined on a Separate Sample.*—Weigh 1.00 g. of the finely ground, dried sample into a platinum crucible, add 10 drops perchloric acid, $HClO_4$, and 20 ml. of HF and heat almost to dryness on a hot plate. Add 5 to 10 ml. of $HClO_4$ and heat until residue has dissolved (Note). Cool, place crucible in a 400-ml. beaker and add 100 ml. of water and heat to boiling. Any residue present, other than silica, should be filtered off and fused with $K_2S_2O_7$ in a porcelain crucible and added to the main solution.

NOTE.—Decomposition of raw fireclays and some fired refractories can be effected equally as well by substituting an equal volume of H_2SO_4 (1:1) for the $HClO_4$. In this case, take to fumes once, cool, dilute, filter, and fuse any residue remaining undissolved.

(*c*) Determine iron using one of the approved methods referred to in the Preface.

Titania

12. Determine TiO_2 colorimetrically in the 25-ml. portion reserved for this purpose (Section 11) by oxidizing both the sample and the standard titania solution with several drops of a H_2O_2 solution (30 per cent). Compare the colors either in Nessler tubes or in a suitable colorimeter. Use a H_2SO_4 solution (5 per cent) for diluting purposes in matching the colors.

Alumina

13. Subtract the calculated weight of Fe_2O_3 (Section 11 (*a*) or (*b*)), TiO_2 (Section 12), and SiO_2 (Section 9) from the weight of R_2O_3 (Section 10)

and the remainder is the weight of Al_2O_3 plus small amounts of the oxides which may include those previously mentioned in Section 9. These are generally considered as Al_2O_3 in reporting the analysis of fireclay refractories.

Lime

14. Evaporate the filtrate reserved (Section 10) for the determination of CaO and MgO to about 200 ml., add 10 to 15 ml. of a saturated ammonium oxalate $(NH_4)_2C_2O_4$ solution, and 2 to 3 ml. of NH_4OH. Heat for 1 to 2 hr., by which time the volume should be about 75 to 100 ml. Allow the precipitated calcium oxalate to settle. Decant through a dense paper (Whatman No. 40 or equivalent), taking care to retain the precipitate in the beaker, wash several times with warm water by decantation, and then wash the paper until free from salts. Reserve the filtrate for the MgO determination (Section 15). Return the paper to the beaker containing the precipitate, add 100 ml. of a H_2SO_4 solution (5 per cent), warm and titrate to a faint pink end point with standard 0.04 N $KMnO_4$ solution. A blank should be previously determined for the effect of the paper.

NOTE.—For greater accuracy a double precipitation should be made, in which case, after precipitating the calcium oxalate as described above, decant the liquid and wash the beaker and paper several times with warm water. Dissolve the precipitate on the paper with warm HCl (1:4), allowing it to run into the beaker containing the major portion of the calcium oxalate. Wash the paper with hot water. To the solution (about 75 to 100 ml. in volume) add several milliliters of saturated $(NH_4)_2C_2O_4$ solution and ammonia in slight excess. Heat for 2 hr., filter, wash, and titrate as described above.

Magnesia

15. Evaporate the filtrate from the CaO determination (Section 14) to about 150 to 200 ml. and add 2 to 3 g. of diammonium phosphate $(NH_4)_2HPO_4$, stir until dissolved and then add NH_4OH until alkaline and then 20 ml. in excess. Allow the solution to stand overnight. Filter, wash with NH_4OH solution (5 per cent). Dissolve the precipitate on the paper with hot HCl (1:4), allowing it to run into the beaker containing the precipitate. Wash the paper with hot water. To the solution, which should be not more than 100 ml. in volume, add 0.1 to 0.2 g. of $(NH_4)_2HPO_4$. Make ammoniacal and then add a slight excess with constant stirring until the precipitate is well formed, then add 10 ml. more of NH_4OH and allow to stand overnight or at least for 4 hr. Filter through a No. 40 Whatman paper or equivalent. Transfer the precipitate to the paper and wash well with a NH_4OH solution (5 per cent). Place the paper in a weighed platinum crucible and burn off the paper at a low temperature (below 900 C.), and ignite to constant weight at 1050 to 1100 C. (15 to 30 min. is sufficient).

Alkalies[1]

16. (*a*) Weigh out 1.00 g. of the sample (ground to an impalpable powder) and 3.0 g. of NH_4Cl into an agate mortar and mix well. Add

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[1] This procedure for the determination of alkali is the J. Lawrence Smith method.

7 to 8 g. of $CaCO_3$ and again mix intimately. Place a $\frac{1}{8}$-in. layer of $CaCO_3$ in the bottom of a platinum crucible, and then add the above mixture, tapping the crucible occasionally to obtain a dense mass. Place a $\frac{1}{8}$-in. layer of $CaCO_3$ on the top. Heat the crucible over a low flame until ammonia fumes are no longer given off, then increase the heat so that the bottom half of the crucible is a dull red, and maintain this temperature for about 1 hr. Cool, fill the crucible three-fourths full of water and heat until the contents can be taken out and crushed in an agate mortar. Transfer to a platinum or porcelain dish by means of a jet of water. Evaporate to a low volume, decant through a No. 40 Whatman paper, or equivalent, and wash the material in the dish several times by decantation with warm water. Transfer to the paper and wash several times with hot water. Acidify with several milliliters of HCl and evaporate to a volume of 150 to 200 ml. Add several milliliters of NH_4OH and sufficient $(NH_4)_2CO_3$ to precipitate the lime, keeping the dish covered with a watch glass. Warm until the precipitate settles out. Filter and wash with warm water When the solution has reached a low volume, add a small lump of $(NH_4)_2CO_3$ to determine if practically all calcium has been precipitated. If no precipitate forms, evaporate to dryness, otherwise precipitate and filter as before. Drive off the ammonium salts by heating just short of a dull red. Dissolve the residue in water and add a few milliliters of a saturated solution of $(NH_4)_2CO_4$ and 1 to 2 ml. of NH_4OH to precipitate the last trace of calcium. Heat for 30 to 45 min., filter and wash with water containing $(NH_4)_2C_2O_4$ (0.1 per cent). Catch the filtrate in a weighed platinum dish. Add several drops of HCl and evaporate to dryness. Ignite gently as before and weigh as NaCl and KCl.

(*b*) The separation of the potassium and sodium must be carried out in an atmosphere free from ammonia fumes. Add to the solution of the combined chlorides in a small porcelain dish, sufficient chloroplatinic acid solution to react with all of the sodium and potassium. The necessary amount to use is readily calculated from the known strength of the platinum solution and the weight of mixed chlorides counted as sodium chloride. The dilution of the resulting solution should be such that when heated on the steam bath any precipitate that may have formed entirely dissolves. This is to prevent inclusion of mother liquor in a mass of crystals suddenly formed. Evaporate until the solution is just syrupy enough to solidify on cooling. Do not evaporate to dryness, as this will dehydrate the sodium salt and render it less soluble in alcohol. Drench the residue with a solution of alcohol (80 per cent), filter by decantation through a small paper, wash by decantation with more of the alcohol, crushing the crystals with a small pestle or widened and rounded end of a short glass rod. Reserve the filtrate and washings if sodium is to be determined directly. The residue should be golden yellow. An orange red color indicates incomplete removal of the sodium salt. It is unnecessary to bring the mass of the precipitate upon the filter. Dry dish and paper for a few moments, to remove adhering alcohol. Dissolve the precipitate on the filter, with hot water, catching the solution in a weighed crucible or small dish of platinum. Evaporate to dryness and add the salt that is still in the porcelain dish. If the salt is at all lumpy, redissolve it in water and again evaporate to dryness. Heat for 1 hr. at 130 C. in an air oven (100 C. suffices for very small amounts of fine-grained precipitate). It is necessary to cover the receptacle at first because the precipitate is prone to decrepitate. When dry, cool, and weigh as K_2PtCl_6. Calculate the oxides, as follows:

$$
\begin{aligned}
KCl &= \text{Weight of } K_2PtCl_6 \times 0.3068 \\
K_2O &= \text{Weight of } K_2PtCl_6 \times 0.1941 \\
NaCl &= \text{Total chlorides} - KCl \\
Na_2O &= NaCl \times 0.5303
\end{aligned}
$$

SILICA REFRACTORIES

Special Solutions and Reagents Required

17. (*a*) *Sulfuric Acid (25 per cent by volume).*

(*b*) *Nitric Acid (50 per cent by volume).*

(*c*) *Standard Potassium Permanganate Solution (0.01 N).*—Dissolve 0.325 g. in 1000 ml. of water. Standardize against the National Bureau of Standards' standard sample 40*c* of sodium oxalate.

Moisture

18. Weigh 1.00 g. of the sample and heat at a temperature not under 105 nor over 110 C., to constant weight.

Loss on Ignition

19. Remove hygroscopic moisture by heating the air-dried sample for 1 hr. at 105 to 110 C. Then weigh 2.00 g. of the dried sample and heat to constant weight over a blast lamp, or in an electric furnace at 900 to 1000 C. Record the loss in weight as the ignition loss.

Silica

20. Transfer 1.0000 g. of the moisture-free sample, or the equivalent of the ignited residue from the loss on ignition determination (Section 19) to a 30-ml. platinum crucible. Add 10 ml. of HNO_3 (1:1) and 5 ml. of HF. Cover the crucible, place on a hot plate on an asbestos pad, and heat. Heat gently at first, gradually increasing the temperature. The final temperature should be regulated to prevent boiling or spattering. Heating in this manner for 1 hr. is sufficient to decompose the sample Rinse the underside of the crucible lid with a few drops of water, allowing it to run into the crucible. Evaporate the crucible contents to dryness. Cool, add 10 ml. of HNO_3 (1:1) and again evaporate to dryness. Introduce the acid slowly by pouring down the sides of the crucible. Evaporate three times

with the HNO_3 to insure complete volatilization of the fluorides. Ignite gently at first and then finally to constant weight at about 1200 C. Cool in a desiccator, and weigh. The loss in weight minus the loss on ignition (Section 19) represents all but a possible trace of the SiO_2 in the sample

Iron and Aluminum Oxides

21. Fuse the residue in the crucible from the silica determination (Section 20) by heating gently with about 2 g. of potassium pyrosulfate, $K_2S_2O_7$, or sodium pyrosulfate, $Na_2S_2O_7$, cool, dissolve in water and acidify with 3 to 4 ml. of HCl. Dilute to 150 to 200 ml., add 5 g. of NH_4Cl as well as 3 drops of methyl red solution (0.1 per cent). Heat to boiling, add NH_4OH (1:1) slowly, finally drop-by-drop, until the solution is just alkaline to the indicator. Boil for 1 to 2 min. and filter through a 9-cm. rapid filter. Do not wash. Reserve the filtrate. Transfer the paper and precipitate to the beaker in which the precipitation was made, add 50 ml. of HCl (1:4) and digest covered on the hot plate, shaking frequently until the precipitate is dissolved and the paper well macerated. Dilute, add methyl red, reprecipitate with NH_4OH (1:1), boil, and filter, exactly as before. Add this second filtrate to the first filtrate for the subsequent determination of lime (Section 25). Swab the beaker and rod and wash the paper and precipitate with NH_4Cl solution (2 per cent). Dry, then ignite the paper and precipitate in a tared platinum crucible of 30-ml. capacity at 1000 to 1100 C. to constant weight with access to air. Cool in a desiccator, and weigh as R_2O_3 which is mainly the iron and aluminum oxides. To recover the trace of silica, which may have remained with the R_2O_3 group, add 2 drops of H_2SO_4 (1:4) and 1 ml. of HF to the crucible and evaporate to dryness. Cover crucible and ignite at about 1100 C. to constant weight. Cool and weigh. The loss in weight represents additional silica which should be added to that determined in Section 20.

Iron Oxide

22. Transfer 1.00 g. of the dried sample to a 30-ml. platinum crucible and moisten with a few drops of water. Add 3 ml. of HNO_3, and about 15 ml. of HF. Evaporate at a moderate heat to dryness, taking care not to allow any loss by spattering. Decompose the nitrates and fluorides by ignition at about 1000 C. for 15 min. Now add to the residue in the crucible a small piece of $K_2S_2O_7$, or $Na_2S_2O_7$, and fuse. Dissolve the fusion in 25 ml. of HCl (1:1) and heat to boiling. Reduce the iron by adding $SnCl_2$ solution, drop by drop from a pipette with constant swirling of the beaker, until the solution is colorless, then add 1 drop in excess. Cool quickly in running water, then add at one stroke 15 ml. of saturated $HgCl_2$ solution. Allow to stand for 3 min., transfer with washing to a 1000-ml. beaker containing 300 ml. of cold distilled water and 25 ml. of $MnSO_4$ solution. Titrate with standard 0.01 N $KMnO_4$ solution, added very slowly while stirring, until a permanent pink end point is obtained.

Titania

23. Fuse the ignited R_2O_3 residue after recovery of SiO_2 (Section 21) with a very small piece of $K_2S_2O_7$ or $Na_2S_2O_7$. Cool and dissolve the fusion by the addition of about 25 ml. of warm water and 1 to 2 drops of H_2SO_4 (1:1) to the crucible. It is important at this point to keep the solution to a small volume. Transfer to a 50-ml. volumetric flask, add a few drops of H_2O_2 and dilute to the mark. Place the 50 ml. of solution in a small Nessler tube. Compare the color of this solution with the color of a known standard solution. A satisfactory solution is one of such strength that 1 ml. equals 0.0001 g. of TiO_2. To make the comparison use any standard colorimeter, or place an appropriate amount of standard solution in a second Nessler tube and dilute with water from a burette until the color is matched. From the amount of water added, calculate the percentage of TiO_2 in the sample.

Alumina

24. Subtract the calculated weight of Fe_2O_3 (Section 22), the weight of SiO_2 recovered (Section 21), and the calculated weight of TiO_2 (Section 23) from the weight of R_2O_2 (Section 21) and the remainder is the weight of the Al_2O_3, plus small amounts of oxides of any other elements that were precipitated.

NOTE.—These may include P_2O_5 and ZrO_2, but they are present in such small amounts as to be disregarded in the complete analysis of silica refractories.

Lime

25. Acidify the combined filtrate from the R_2O_3 precipitation with several milliliters of HCl (1:1) (Section 21) and evaporate to a volume of about 250 ml. Heat to incipient boiling, add 25 ml. of a saturated $(NH_4)_2C_2O_4$, solution and NH_4OH until slightly alkaline. Stir thoroughly and allow the precipitate to settle out, keeping the solution warm for 3 to 4 hr. but not boiling. Filter and wash five times with cold water. Save the filtrate. Dissolve the washed oxalate precipitate with 50 ml. of diluted HCl (1:1), dilute to about 150 ml. and precipitate as before. Wash the beaker, precipitate, and paper with cold water until $(NH_4)_2C_2O_4$ is removed. Avoid excessive washing. Combine the filtrates for the magnesia determination (Section 26). To the beaker used for the precipitation, add 150 ml. of a H_2SO_4 solution (5 per cent), then introduce the paper containing the precipitate. Heat to almost boiling and titrate with 0.1 *N* $KMnO_4$ solution. A blank should be previously determined for the effect of the paper.

Magnesia

26. To the combined filtrates from the lime determination (Section 25), which have been reduced by evaporation to about 300 ml., add 2 to 3 g. of $(NH_4)_2HPO_4$. Stir until dissolved and then add NH_4OH slowly until alkaline, and then 30 ml. of NH_4OH in excess. Allow the solution to stand for at least 4 hr., and preferably overnight if the total amount of solution exceeds 400 ml. in volume. Filter, wash with diluted NH_4OH solution

(5 per cent). Dissolve from the paper with hot HCl (1:1) and reprecipitate by adding 0.1 g. of $(NH_4)_2HPO_4$, diluting, rendering ammoniacal and letting stand as before. After filtering and washing as before, char the paper and burn off the carbon below 900 C., and then ignite in an electric muffle furnace at about 1100 C. to constant weight. The ignited material consists of magnesium pyrophosphate, $Mg_2P_2O_7$. Calculate the weight of MgO by multiplying the $Mg_2P_2O_7$ by 0.3621.

Alkalies

27. Determine the alkalies by the procedure described in Section 16 (*a*) and (*b*) for the analysis of fireclay refractories.

HIGH-ALUMINA REFRACTORIES

(*Includes Bonded Refractories made from Diaspore, Fused Alumina, and the Sillimanite Minerals, as well as Electrocast.*)

Special Solutions Required

28. (*a*) *Potassium Nitrate (1 per cent).*—Dissolve 1 g. in 100 ml. of water.

(*b*) *Molybdate Reagent.*—Mix 100 g. of pure molybdic anhydride or 118 g. of molybdic acid (85 per cent) with 400 ml. of water, add 80 ml. of NH_4OH, and filter when solution is complete. Prepare a second solution containing 400 ml. of HNO_3 and 600 ml. of water. Agitate the HNO_3 solution by means of a current of air and add the molybdate solution very slowly. Continue agitation for 1 hr. Add a small crystal of $(NH_4)_2HPO_4$, let stand over night, filter and keep in a glass-stoppered bottle.

(*c*) *Standard Sodium Hydroxide Solution.*—Dissolve 60 g. of NaOH in 100 ml. of distilled water in a 150-ml. test tube of resistant glass. Stopper tightly with a cork covered with tin foil and let stand until the supernatant liquid is clear. By means of a pipette take out 5.0 ml. of the clear solution and dilute to 1 liter with freshly boiled water.

(*d*) *Standard Nitric Acid Solution.*—Dilute 5.0 ml. of HNO_3 (sp. gr. 1.42) to 1 liter with freshly boiled water.

NOTE.—Adjust these solutions until they exactly agree and standardize against National Bureau of Standards' standard sample No. 78 of known phosphorous content.

Moisture

29. Weigh 1.00 g. of the sample and heat at a temperature not under 105 nor over 110 C., to constant weight.

Loss on Ignition

30. Remove hygroscopic moisture by heating the air-dried sample for 2 hr. at 110 to 120 C. Then weigh 1.000 g. of the dried sample and heat to constant weight over a blast lamp, or in an electric furnace at 1000 C. Record the loss in weight as the ignition loss.

Silica

31. (*a*) Weigh 1.0000 g. of the sample into a platinum crucible which is free from any iron stain. Add 0.40 g. of anhydrous borax, $Na_2B_4O_7$, and 2.00 g. of Na_2CO_3. Mix well and fuse first at a low red heat, gradually increasing to the full temperature of a blast burner. Blast 15 min. Dissolve the melt in diluted HCl (1:4) in a porcelain evaporating dish, add 2 to 3 ml. of diluted H_2SO_4 (1:1) (Note 1) and evaporate to dryness. Wash down the sides of the dish with 25 ml. of methyl alcohol (Note 2) saturated with HCl gas. Evaporate, add methyl alcohol solution and evaporate again. Bake to dehydrate the silica. Cool, add 15 ml. of HCl and 50 ml. of water, boil 1 to 2 min. and filter on a No. 40 Whatman or similar filter paper. Wash four to five times with hot diluted HCl (1:4) solution, then seven to eight times with hot water. Reserve the paper containing the silica precipitate. Transfer the filtrate to the original dish and evaporate to dryness and bake. Add 15 ml. of HCl and 50 ml. of water, bring just to a boil, filter on a No. 42 Whatman or similar filter paper and wash as before. Reserve the filtrate.

NOTE 1.—Diluted H_2SO_4 is used to prevent TiO_2 from hydrolizing and precipitating with the silica.

NOTE 2.—Methyl alcohol is used to eliminate traces of boron.

(*b*) Place the two filter papers containing the SiO_2 precipitate in a platinum crucible, ignite carefully until the paper is burned off, then heat at 1100 to 1200 C. for 10 to 15 min. Cool, weigh and repeat heating at 1100 to 1200 C. until constant weight is obtained. Moisten the ignited precipitate with water, add HF slowly until the crucible is half-full, then add 5 drops of H_2SO_4 and evaporate to fumes. Cool, add 2 to 3 ml. of HF and 2 drops of H_2SO_4 and evaporate to dryness. Heat the crucible to bright redness and blast at 1100 to 1200 C. for 5 min. Cool, weigh, and repeat blasting until constant weight is obtained. The loss in weight represents the SiO_2 content except for the small amount which is later recovered from the alumina precipitate.

Separation of Al_2O_3 and P_2O_5 from Fe_2O_3, TiO_2, CaO, and MgO

32. Fuse the residue from the HF evaporation (Section 31 (*b*)) with a little $K_2S_2O_7$, dissolve the melt in a diluted HCl solution (1:4) and add to the filtrate from the silica precipitate which was reserved. Reduce the volume to about 75 ml. and nearly neutralize with NaOH solution (10 per cent). Slowly and with constant swirling pour into a 250-ml. volumetric flask containing 100 ml. of a NaOH solution (10 per cent), to which has been added 1 g. each of Na_2CO_3 and Na_2O_2 (Notes 1 and 2). Warm on a water bath for 1 hr. and cool to room temperature. Dilute with water to the mark, mix well, and let stand a few minutes. Filter off the precipitate on a filter paper which has been previously washed with a NaOH solution (10 per cent) and dried, catching the filtrate in a dry beaker. Do

not wash the precipitate. From the filtrate take a 100-ml. aliquot for the determination of Al_2O_3 (Section 36) and another 100-ml. aliquot for the determination of P_2O_5 (Section 35).

NOTE 1.—The Na_2CO_3 is used to precipitate calcium and magnesium along with iron and titanium.

NOTE 2.—The Na_2O_2 is used to oxidize any chromium present so that it may later be separated from the Al_2O_3.

Iron Oxide

33. Dissolve the precipitate on the filter paper and that adhering to the flask with a hot diluted HCl solution (1:4). Adjust the volume to about 100 ml., add 5 g. of NH_4Cl and heat to boiling. Add NH_4OH while stirring slowly until there is a slight excess. Again heat to boiling, filter hot, and wash five times with a hot NH_4Cl solution (2 per cent). Place the paper containing the precipitate in the original beaker, add 15 ml. of HCl, stir to macerate the paper, dilute with hot water to 100-ml. volume, and precipitate with NH_4OH. Filter, and wash as before. Combine the two filtrates and reserve for the determination of CaO and MgO. Dissolve the precipitate in hot diluted HCl (1:4) and cool. Pour into a 100-ml. volumetric flask, dilute with water to the mark and mix. Draw off exactly 5.0 ml. of the solution and reserve for the determination of TiO_2 (Section 34). Pour the remaining solution into a 250-ml. beaker and evaporate to dryness. Add 25 ml. of HCl (1:1) and heat to boiling. From this point proceed in accordance with the procedure described in Section 22 for the determination of Fe_2O_3 in the analysis of silica refractories.

Titania

34. Add to the 5.0-ml. portion previously reserved (Section 33), 10 ml. of H_2SO_4 (1:1) and evaporate to fumes. Dilute with 10 to 20 ml. of water and cool. Pour into a 100-ml. volumetric flask, add a few drops of H_2O_2 (30 per cent), dilute to the mark and mix. To a 100-ml. volumetric flask, add exactly 20.0 ml. of the standard TiO_2 solution and 10 ml. of H_2SO_4 (1:1) solution. Add a few drops of H_2O_2 solution (30 per cent), dilute to the mark and mix. Compare the colors either in Nessler tubes or in a suitable colorimeter.

Phosphorous Oxide

35. Acidify the 100-ml. aliquot portion reserved for this determination with HNO_3. Dilute to 200-ml. volume and precipitate the Al_2O_3 and P_2O_5 by adding a slight excess of NH_4OH solution. Filter, wash a few times with hot water, and dissolve the precipitate in 100 ml. of diluted HNO_3 (1:9). Transfer to a 300-ml. Erlenmeyer flask, heat to boiling and add a saturated solution of $KMnO_4$ until a pink color persists. Decolorize with a few drops of sulfurous acid, add 15 ml. of HNO_3, cool, adjust the volume to about 125 ml. and add 20 ml. of NH_4OH, and 40 ml. of molybdate

reagent. Shake for 10 min., let stand for 30 to 40 min., filter and wash the flask, paper and precipitate at least ten times with KNO_3 solution (1 per cent). Return the paper and precipitate to the flask. Add an excess of standard NaOH solution and 25 ml. of water (both free from CO_2). Shake until the precipitate is dissolved and dilute to a 100-ml. volume. Add 2 to 3 drops of phenolphthalein solution (0.2 per cent), then titrate with standard HNO_3 to the disappearance of the pink color. Subtract from the original NaOH added, the volume equivalent to the HNO_3 required to discharge the color. The difference gives the milliliters of NaOH equivalent to the P_2O_5 present. Calculate the percentage of P_2O_5.

Alumina

36. (*a*) Dilute the 100-ml. aliquot, previously reserved (Section 32), to a 250-ml. volume and add 5 g. of NH_4Cl. Heat to boiling, add NH_4OH very slowly, with constant stirring, until there is a slight excess as shown by methyl red. Boil 1 to 2 min., filter hot and wash five to six times with hot NH_4Cl solution (2 per cent). Place the paper containing the precipitate in the original beaker, add 15 ml. of HCl and, with the aid of a glass stirring rod, macerate the paper. Add boiling water to make up to a volume of about 250 ml. Repeat the precipitation with NH_4OH and filter as before. Wash thoroughly with hot NH_4Cl solution (2 per cent), using at least 250 ml. of wash solution.

(*b*) Place the paper containing the precipitate in a platinum crucible which has been previously weighed with its cover. Carefully burn off the paper, blast at not less than 1200 C. for 10 min., cool, and weigh. Repeat blasting and weighing until constant weight is obtained. The increase in weight is the Al_2O_3, and P_2O_5 plus a little SiO_2 which has escaped precipitation. To obtain the percentage of Al_2O_3, subtract the P_2O_5 (Section 35) and SiO_2 (Section 36 (*c*)).

(*c*) To determine the SiO_2, fuse the precipitate with about 5 g. of $K_2S_2O_7$. Dissolve the melt in warm water, add about 30 ml. of diluted H_2SO_4 (1:1) solution. Evaporate to copious fumes of H_2SO_4 and cool. Take up with water, filter off, wash, ignite in a platinum crucible, and weigh. Treat the residue with 5 ml. of HF and 2 to 3 drops of H_2SO_4, evaporate to dryness, ignite, and weigh. The loss in weight is SiO_2. Add to the original silica determination.

Lime

37. Determine CaO in the filtrate from the Fe_2O_3 precipitate according to the method described for the determination of lime under analysis of fireclay refractories (Section 14).

Magnesia

38. Determine MgO by the method described for the determination of magnesia under analysis of fireclay refractories (Section 15).

Alkalies

39. Proceed as in the determination of alkalies under the analysis of fireclay refractories (Section 16 (*a*) and (*b*)) but with the following precautions:

(*a*) Fuse the sample at 1200 C. to obtain complete decomposition of the material.

(*b*) Cool the top of the crucible to prevent any loss of alkalies by volatilization. Cooling can be accomplished more easily if a platinum crucible of the J. Lawrence Smith, long type is used.

CHROME ORES AND CHROME BRICK

A. Procedure for Complete Analysis

Preparation of Sample for Analysis

40. The sample shall be secured after the manner of any of the approved methods of sampling. If from ores or chrome-bearing refractories, the sample shall be not less than 10 lb., in small nut-size particles when sent to the laboratory. If from chrome brick, the sample shall consist of a small portion from the interior of from 15 to 25 bricks. This 10-lb. sample shall be crushed to pass a No. 20 sieve[1] and quartered until a sample of 100 g. is secured, which shall be ground to pass a No. 100 sieve.[1] From this portion, a smaller working sample shall be quartered and ground in an agate mortar to a very nearly gritless powder. In the case of hard burned brick or hard lump chrome ore the very fine grinding assists materially in solution of the sample.

Methods of Analysis

41. (*a*) Dry the fine, ground powder at 110 C. for not less than 1 hr. Cool in desiccator. Fuse 0.5 g. of the thoroughly dried sample in approximately 10 g. of fused potassium bisulfate in a platinum crucible. The usual cautions of a bisulfate fusion should be observed, that is, the flame turned very low so that the bisulfate just melts, increasing the flame so that the bisulfate remains in a liquid condition, very gradually increasing until the crucible reaches a dull red heat. The object of the slow heating is to retain the sulfuric acid, which is the effective part of the flux. The most refractory ores should be thoroughly fused in 1½ to 2 hr. Remove the crucible from the burner and allow to cool in the air. The bisulfate melt will shrink from the crucible and may be removed easily in one cake. Dissolve the fusion in 200 ml. of hot diluted HCl (1:1), boiling until the solution acquires a clear, bright green color. Transfer the solution to a porcelain evaporating dish and evaporate to dryness on a water bath. Bake at exactly 120 C. until free from hydrochloric acid fumes. Moisten with hot diluted HCl (1:1), evaporate on a water bath and bake a second time at 120 C. Dissolve in 200 ml. of hot diluted HCl (1:1), and filter out the

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[1] For detailed specifications for these sieves, see the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials, 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

silica. Ignite and weigh the silica in a platinum crucible. Add two drops of concentrated H_2SO_4 and sufficient HF to remove all silica. Evaporate, ignite and weigh. The difference gives the amount of silica present in the sample. Should there be any appreciable residue left in the crucible it should be fused with a small amount of fused $KHSO_4$ and added to the filtrate from the silica.

(*b*) Dilute the filtrate from the silica to about 500 ml. and precipitate iron, aluminum, chromium and titanium, if present, with NH_4OH, using care to have not more than two drops of NH_4OH in excess in order to avoid solution of chromium and aluminum hydroxide in the alkaline solution. On account of the relatively large amount of magnesium that is usually present in chrome ores and chrome brick it is advisable to dissolve the precipitate in hot diluted HCl and precipitate a second time. Filter out the combined hydroxides and dissolve them on the filter paper with HNO_3, catching the solution in a porcelain casserole. Add a few crystals of $KClO_3$ and evaporate the nitric solution, adding a crystal of $KClO_3$ from time to time until the chromium is completely oxidized, which is indicated by a change from a dull green to a brilliant orange color. Dilute this solution with hot water and again precipitate with NH_4OH, filtering as before. The filtrate contains a large percentage of the chromium, while the residue on the filter consists of iron and aluminum hydroxides and titanium, if present. Repeat the oxidation two or three times until the filtrate from the iron and aluminum hydroxide precipitation shows no further traces or chromium. Concentrate and combine the filtrates containing chromium to approximately 500 ml. Add H_2SO_3 until the liquid changes from a bright orange to a brilliant green color and heat to boiling. Add NH_4OH to precipitate chromium as hydroxide, having not more than two drops in excess, as stated above. Filter, wash well with hot water, ignite and weigh as Cr_2O_3. Ignite the iron and aluminum and titanium hydroxides in a platinum crucible and weigh. Fuse the combined oxides with a small amount of $KHSO_4$, following the same precautions as mentioned above. Dissolve the fusion in diluted H_2SO_4, reduce with H_2S and titrate the iron with standard permanganate solution. Determine the titanium present by the colorimetric method, as described in Section 12. Subtract the combined amounts of Fe_2O_3 and TiO_2 from the total weight of the three oxides and thus determine Al_2O_3. If titanium is absent, reduce the iron with metallic zinc and titrate with $KMnO_4$, which is a little quicker method than the H_2S reduction.

(*c*) To the combined filtrates from the precipitations of iron, aluminum, chromium, and titanium hydroxides add bromine, allow to stand for a few minutes and then add sufficient NH_4OH to precipitate the manganese hydroxide. Boil to concentrate the manganese hydroxide if any be present.

Filter, ignite and weigh as Mn_3O_4. In the filtrate from the manganese add NH_4OH and $(NH_4)_2C_2O_4$. Heat to boiling and when the calcium oxide has settled, filter with the usual precautions. The calcium oxide may be determined by the volumetric method. (Refer to the determination of calcium oxide as described in Section 14.)

(*d*) Evaporate the filtrate from the calcium oxalate to dryness with HNO_3 to remove the excess of ammonium salts. Dissolve the residue in hot water, filter, allow to cool and determine MgO as described in Section 15.

(*e*) Determine alkalies by the method described in Section 16.

B. *Rapid Routine Method*

Preparation of Sample for Analysis

42. The sample shall be secured after the manner of any of the approved methods of sampling. If from ores or chrome bearing refractories the sample shall not be less than 10 lb. in small nut-sized particles when sent to the laboratory. If from chrome brick, the sample shall consist of a small portion from the interior of from 15 to 25 bricks. This 10-lb. sample shall be crushed to pass a No. 20 sieve[1] and quartered, until a 4-oz. sample is secured, which shall be ground to pass a No. 100 sieve.[1] From this portion a smaller working sample shall be quartered and ground in an agate mortar to a very nearly gritless powder. In the case of hard burned brick or hard lump chrome ore the very fine grinding assists materially in solution of the sample. Dry the fine, ground powder at 110 C. for not less than 1 hr. Cool in a desiccator.

Chromium Oxide

43. Fuse 0.5 g. of the sample in 8 to 10 g. of sodium peroxide in a nickel or porcelain crucible over a bunsen burner, being careful not to heat it too strongly. When fused, leach out with cold water, boil 15 min. to decompose any sodium peroxide. Cool and add 100 ml. of diluted H_2SO_4 (1:1). Cool to tap-water temperature and titrate with ferrous ammonium sulfate and potassium permanganate.

Silica and Iron Oxide

44. Fuse 0.5 g. of the sample in a nickel crucible over a bunsen burner. Leach out with cold water, make acid with HCl and evaporate to dryness. Take up with 200 ml. of water and add 10 ml. of HCl. Filter off silica and wash well with hot water and hot diluted HCl. For high silica a second evaporation is necessary. The filtrate or combined filtrate is made alkaline with sodium hydroxide and about 5 ml. of bromine added. The mass is allowed to digest for 30 min. on a steam bath. This oxidizes the iron and

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[1] For detailed specifications for these sieves, see the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials, 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

chromium, precipitates the iron and dissolves the chromium and aluminum. After precipitating the iron twice it is filtered off on an asbestos plug and washed well with hot water. This precipitate of iron may be dissolved off the plug with hot diluted HCl (1:1) and determined by the stannous chloride reduction, and permanganate method (Section 11).

(*b*) A blank should be run in conjunction with this method and deductions made when necessary to determine the purity of the chemicals used.

MAGNESITE REFRACTORIES

Special Solutions Required

45. The following solutions will be required:

(*a*) *Hydrochloric Acid (2.5 per cent by volume).*

(*b*) *Nitric Acid (30 per cent by volume).*

(*c*) *Nitric Acid (3 per cent by volume).*

(*d*) *Ethyl Alcohol (Absolute).*—Certain commercial brands of denatured absolute alcohol are satisfactory as well as being considerably less expensive than the c.p. absolute alcohol.

(*e*) *Potassium Permanganate Solution (Approximately 0.5 N).*—Dissolve 16 g. of $KMnO_4$ in 1000 ml. of water.

(*f*) *Ammonium Chloride-Hydrochloric Acid Solution.*—Dissolve 50 g. of NH_4Cl in water, add 50 ml. of HCl (sp. gr. 1.19) and dilute to 1000 ml.

(*g*) *Standard Sodium Arsenite Solution.*—Dissolve 0.908 g. of pure arsenious oxide, As_2O_3, in a small amount of hot Na_2CO_3 solution, cool, filter and dilute to 1 liter. Standardize against a steel of known manganese content.

Preparation of Samples for Analysis

46. A 10-lb. sample carefully secured after the manner of one of the approved methods of sampling shall be crushed to pass a No. 8 sieve,[1] mixed thoroughly, and reduced by quartering to about 0.5 lb. This shall be crushed to pass a No. 20 sieve,[1] mixed and quartered to about 50 g. This 50-g. sample shall then be ground in an agate mortar so that it will all pass a No. 100 sieve,[1] mixed thoroughly and placed in an air-tight container until analyzed, since finely ground, dead-burned magnesite gradually absorbs and fixes moisture and carbon dioxide.

Moisture

47. Heat 2.0 g. of the sample in a platinum crucible for 1 hr. at 105 to 110 C. Record the loss in weight as hygroscopic moisture.

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[1] For detailed specifications for these sieves, see the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials, 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

Loss on Ignition

48. Using the sample from which the hygroscopic moisture has been removed (Section 47), heat it to constant weight over a blast lamp or in an electric furnace at 900 to 1000 C. Record the loss in weight as the ignition loss.

Silica

49. Transfer 1.0000 g. of the moisture-free sample, or the equivalent of the ignited residue from the loss on ignition determination (Section 48), to a 210-ml. casserole. Moisten the solids with a few milliliters of water, slowly add 20 ml. of HCl, and cover the casserole. While covered, digest on the hot plate until solution is complete and all gritty particles have disappeared, breaking up any solid cake from time to time with a stirring rod. Uncover, add 8 ml. of perchloric acid, $HClO_4$ (60 to 70 per cent), and evaporate to heavy white fumes of $HClO_4$. Cover and boil so that acid refluxes for 10 to 15 min. Remove from the hot plate, cool, wet with a few milliliters of water and 5 ml. of HCl, warm for a few minutes on the hot plate, add 50 ml. of hot water, heat to approximately boiling and filter through a 9-cm. rapid filter. Wash the paper and precipitate ten times with a hot HCl solution (2.5 per cent), then three times with hot water. Return the filtrate to the casserole in which the first evaporation was made, add 2 ml. of $HClO_4$ and evaporate to fumes on the hot plate, boiling as before. Cool, take up in acid and water, filter, and wash as before. Reserve the filtrate. Place both papers and precipitates in a tared platinum crucible. Dry, then burn off papers and ignite to constant weight in the muffle or over a moderate blast. Cool in a desiccator and weigh as impure silica. Add two drops of diluted H_2SO_4 (1:1) and 1 ml. of HF. to the impure silica in the crucible. Digest for 5 to 10 min., covered, on the hot plate, then remove the cover and evaporate to dryness. Fume off the H_2SO_4 over a moderate heat and ignite or blast for a few minutes. Cool in a desiccator, and weigh again. Subtract the weight of the crucible and residue from the weight of the crucible and impure silica and the difference is the SiO_2. Fuse the residue in the crucible by heating gently with a small piece of $K_2S_2O_7$ or $Na_2S_2O_7$ about the size of a pea, cool, dissolve in water and add to the reserved filtrate from the silica.

Iron and Aluminum Oxides

50. Add to the filtrate from the silica determination (Section 49), which should be in a 400-ml. beaker, 5 g. of NH_4Cl. Dilute from 150 to 200 ml., add three drops of methyl red solution (0.1 per cent), heat to boiling, add NH_4OH (1:1) slowly; finally drop by drop, until the solution is just alkaline to the indicator. Boil for 1 or 2 min. and filter through a 9-cm. rapid filter. Do not wash. Reserve the filtrate. Transfer the

paper and precipitate to the beaker in which the precipitation was made, add 50 ml. of HCl (1:4) and digest covered on the hot plate, shaking frequently until the precipitate is dissolved and the paper well macerated. Dilute, add methyl red, reprecipitate with NH_4OH (1:1), boil and filter, exactly as before. Add this second filtrate to the first filtrate for the subsequent determination of lime (Section 53 (*a*)). Swab the beaker and rod and wash the paper and precipitate with NH_4Cl solution (2 per cent). Dry, then ignite the paper and precipitate in a tared platinum crucible at 1000 to 1100 C. for 1 hr. with access of air. Cool in a desiccator and weigh as R_2O_3 which is mainly the Fe_2O_3 and Al_2O_3.

Iron Oxides

51. Weigh 2.00 g. of the sample into a 150-ml. beaker and cover. Dissolve by slowly adding HCl, keeping the beaker on the hot plate. Finally, add about 5 ml. of HCl in excess. Heat to gentle boiling until the siliceous residue undissolved appears iron-free. Filter and fuse the residue with a very little $K_2S_2O_7$ or $Na_2S_2O_7$ in a porcelain or vitreous silica crucible to avoid introducing platinum. Dissolve the fused material and add to the filtrate. Add a few drops of a strong solution of $KMnO_4$ (about 0.5 *N*) to oxidize any organic matter that may be present. Boil. When the excess of $KMnO_4$ is decomposed and chlorine fumes have disappeared, reduce the iron by adding $SnCl_2$ solution, drop by drop from a pipette with constant swirling of the beaker, until the solution is colorless, then add 1 drop in excess. Cool quickly in running water, then add at one stroke 15 ml. of saturated $HgCl_2$ solution. Allow to stand for 3 min., transfer with washing to a 600-ml. beaker containing 300 ml. of cold distilled water and 25 ml. of $MnSO_4$ solution. Titrate with standard 0.1 *N* $KMnO_4$ solution, added very slowly while stirring, until a permanent pink end point is obtained.

Alumina

52. Subtract the calculated weight of iron oxide, Fe_2O_3 (Section 51) from the weight of R_2O_3 (Section 50) and the remainder is the weight of the Al_2O_3 plus small amounts of oxides of any other elements that were precipitated (Note).

NOTE.—These may include P_2O_5, TiO_2 and small amounts of SiO_2, but they are present in such small amounts as to be disregarded in the complete analysis of magnesite refractories.

Lime

53. (*a*) *Method A. When MgO is to be Determined Directly.*—Acidify with several milliliters of HCl (50 per cent) the combined filtrate from the R_2O_3 precipitation (Section 50) and evaporate to a volume of about 250 ml. Heat to incipient boiling, add 25 ml. of a saturated $(NH_4)_2C_2O_4$ solution (about 4 per cent) and NH_4OH until slightly alkaline. Stir thoroughly and allow the precipitate to settle out, keeping the solution warm for 3 to 4 hr.

but not boiling. Filter and wash five times with cold water. Save the filtrate. Dissolve the washed oxalate precipitate with 50 ml. of diluted HCl (1:1), dilute to 250 ml. and precipitate as before. Wash the beaker, precipitate and paper with cold water until $(NH_4)_2C_2O_4$ is removed. Avoid excessive washing. Combine the filtrates for the determination of MgO (Section 54). To the beaker used for the precipitation, add 150 ml. of a H_2SO_4 solution (5 per cent), then introduce the paper containing the precipitate. Heat to almost boiling and titrate with 0.1 *N* $KMnO_4$ solution. A blank should be previously determined for the effect of the paper.

A small part of the lime occasionally escapes the oxalate precipitation and comes down subsequently with the magnesia. To the lime precipitated by the oxalate should accordingly be added that found in the magnesia.

(*b*) *Method B. When MgO is to be Determined by Difference.*[1]—Weigh 2.50 g. of the sample into a 250-ml. beaker, add 10 ml. of water, cover, slowly add HCl until dissolved, stirring frequently and avoiding excessive boiling. Then add about 5 ml. of HCl in excess. Evaporate on a hot plate until very thick and syrupy. At this stage crusts will be forming on the surface and a drop of material placed on the wall of the beaker will immediately solidify. Remove the beaker from the hot plate and allow to partially cool. Add 5 ml. of H_2SO_4 solution (10 per cent). Cover with a watch glass, heat on a hot plate until all soluble matter is dissolved. Cool slightly but not until solids separate. Add 50 ml. of alcohol, stir thoroughly and heat just to boiling on a hot plate. Filter with suction through a Gooch crucible prepared with a moderately thin asbestos mat. If the SiO_2 is less than 3.5 per cent, filter through paper. Rinse the beaker twice with alcohol pouring the washings through the crucible. Drain under suction. Remove the crucible, scrape out the mat with a glass rod into the beaker in which the precipitation of sulfate was made, wash the crucible clean with a hot NH_4Cl - HCl solution, using about 35 ml. If the SiO_2 is high (over 6 per cent) it is advisable to filter it off at this point before precipitating the lime. Dilute to about 150 ml. with hot water, make alkaline with NH_4OH, heat to boiling and precipitate the calcium by slowly adding 15 ml. of a saturated solution of $(NH_4)_2C_2O_4$. Digest on the hot plate just below boiling for 30 min. Filter on a 9-cm. paper, wash with cold water until free from oxalate, testing final washing with a drop of $KMnO_4$ after acidification. Transfer the paper and precipitate to the original beaker, dissolve with 150 ml. of H_2SO_4 (5 per cent) and titrate as in Method A, Section 53 (*a*). Correct for a blank on the paper.

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[1] In this specific procedure very much less lime escapes precipitation and thus the magnesia "by difference" is not subject to this plus error. The procedure further has the advantage that an individual lime determination may be made without separating the other usual constituents.

Magnesia

54. (*a*) Acidify with several milliliters of HCl (50 per cent) the combined filtrates reserved during the determination of CaO (Section 53 (*a*).) Dilute to 500 ml. in a calibrated flask and take a 50 ml. ($\frac{1}{10}$) aliquot portion. Since a 1-g. sample was originally used, this will yield aliquot portions containing between 0.08 to 0.09 g. of MgO. Dilute to 250 ml. and add 2 to 3 g. of diammonium phosphate, $(NH_4)_2HPO_4$. Stir until dissolved and then add NH_4OH slowly until alkaline and then 30 ml. of NH_4OH in excess. Allow the solution to stand for at least 4 hr. and, preferably overnight if the total amount of solution exceeds 400 ml. in volume. Filter, wash with diluted NH_4OH solution (5 per cent). Dissolve from the paper with hot HCl (1:1) and reprecipitate by adding 0.1 g. of $(NH_4)_2HPO_4$, diluting, rendering ammoniacal and letting stand as before. After filtering and washing as before, char the paper and burn off the carbon below 900 C. and then ignite in an electric muffle furnace at about 1100 C. to constant weight. The ignited material consists of magnesium pyrophosphate, $Mg_2P_2O_7$ as well as some of the CaO and MnO. Correction may be made for these as follows:

(*b*) Dissolve the ignited pyrophosphate in a little diluted H_2SO_4 and add enough absolute alcohol to make up 90 to 95 per cent of the final volume. Any calcium present will settle out as $CaSO_4$. Filter, wash with alcohol and dissolve the precipitate in hot diluted HCl. Precipitate with oxalate and titrate with permanganate. Add this CaO to that previously found. The CaO is then calculated to tricalcium phosphate, $Ca_3(PO_4)_2$, by multiplying by the factor 1.845 and this amount deducted from the weight of the $Mg_2P_2O_7$. The correction to the first calcium precipitate is ten times the weight of the CaO precipitated as sulfate.

(*c*) The manganese which contaminates the $Mg_2P_2O_7$ is determined in the alcoholic filtrate from the $CaSO_4$. Evaporate this filtrate until strong fumes are evolved and all organic matter destroyed. Cool, dissolve the residue in 25 ml. of HNO_3 (30 per cent) and determine the manganese by the bismuthate method, as described in Section 55. Any manganese found is calculated to $Mn_2P_2O_7$ by multiplying by the factor 2.585 and this weight deducted from the $Mg_2P_2O_7$.

Manganese as MnO

55. Mix intimately 1.0 to 5.0 g. of the sample (depending upon the amount of manganese expected) with 0.5 to 2.5 g. of anhydrous Na_2CO_3 and ignite in a platinum crucible for 15 min. at about 1000 C. Dissolve the sintered melt in 50 ml. of HNO_3 (30 per cent) and add dropwise sulfurous acid, if there is any evidence of brown hydrated MnO_2. Boil the solution, filter if necessary, cool to room temperature or lower, and add 2 to 3 g. of sodium bismuthate. Agitate the solution for 1 min. and filter through asbestos

(Note). Wash with a HNO_3 solution (3 per cent) until the residue is free from permanganic acid, as determined by the absence of the pink color in the washings. Titrate, at once, the permanganic acid formed, with standard sodium arsenite (1 ml. = 0.0002 g. of Mn). The end point is the disappearance of the pink color. The factor for converting Mn to MnO is 1.291.

NOTE.—A good grade of asbestos, acid washed, and specially prepared for Gooch crucibles, should be used.

TENTATIVE METHODS OF TEST
FOR
COLD CRUSHING STRENGTH AND MODULUS OF RUPTURE OF REFRACTORY BRICK AND SHAPES[1]

A.S.T.M. Designation: C 133 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These methods cover the following tests for refractory brick and shapes:

The cold crushing strength test; and
The modulus of rupture test.

COLD CRUSHING STRENGTH TEST

Apparatus

2. Any form of standard mechanical or hydraulic testing machine may be used for the cold crushing strength test, together with a spherical bearing block with a plane bearing surface the area of which is equal to or greater than the cross section of the test specimen.

Test Specimen

3. The test specimen shall approximate a whole brick in size (9 by $4\frac{1}{2}$ by $2\frac{1}{2}$ in.), and preserve as many of the original surfaces as possible. In preparing specimens from irregular or large refractory shapes, any method involving the use of abrasives, such as high-speed abrasion wheel or rubbing bed, which will produce a specimen with approximately plane and parallel sides, without weakening the structure of the specimen, may be used.

Preparation of Test Specimen

4. (*a*) The specimen to be used for the cold crushing strength test shall be prepared as follows: The two end surfaces ($4\frac{1}{2}$ by $2\frac{1}{2}$ in.) intended to receive the load from the testing machine shall receive a thin coating of shellac and be allowed to dry thoroughly. One of the shellacked bearing surfaces shall then receive a thin coat of a neat mortar of calcined gypsum

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

(plaster of Paris). This mortared face of the specimen shall then be pressed on plate glass or other plane, nonporous surface, which has been previously oiled or covered with waxed paper, and the bedding material allowed to harden. This procedure shall be repeated with the other shellacked surface. Care shall be taken that the opposite faces so coated with bedding material shall be parallel.

(*b*) When the top and bottom surfaces of the specimen are approximately plane and parallel, a cellulose fiber wall board 0.25 in. in thickness and extending 2 in. or more over the edges of the specimen may be substituted for the above method of preparation.

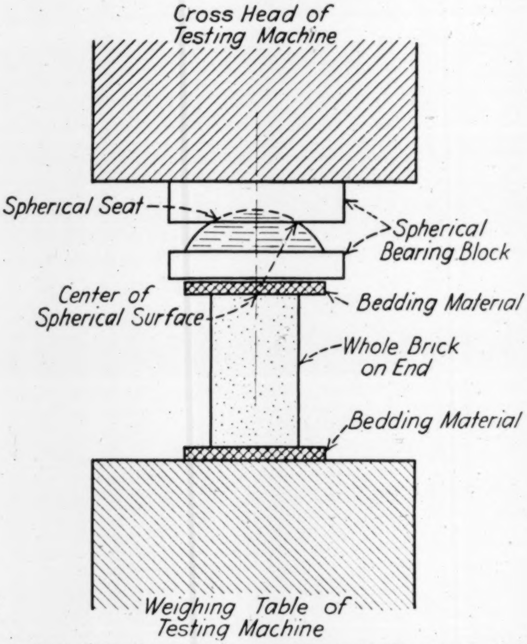

Cross Head of Testing Machine
Spherical Seat
Spherical Bearing Block
Center of Spherical Surface
Bedding Material
Whole Brick on End
Bedding Material
Weighing Table of Testing Machine

FIG. 1.—Recommended Design for Bearing Block for Use in Cold Crushing Test.

(*c*) The report shall state which type of bedding material is used.

Number of Tests

5. Cold crushing strength tests shall be made on at least five brick, or specimens taken from five different refractory shapes.

Cold Crushing Strength Test

6. (*a*) The load shall be applied to the $4\frac{1}{2}$ by $2\frac{1}{2}$-in. ends of the test specimen.

(*b*) The bearing block shall be used on top of the test specimen in vertical testing machines. The center of the sphere of the bearing block shall be used in contact with the top bearing surface of the specimen, and in the vertical axis of the specimen (Fig. 1). The spherical bearing block shall be kept thoroughly lubricated to insure accurate adjustment, which should be made by hand under a small initial load.

(*c*) The speed of head of the testing machine during the application of the load shall not be more than 0.5 in. per min. up to a pressure of 500 lb. per sq. in. Above 500 lb. per sq. in., the speed shall not exceed 0.05 in. per min.

(*d*) During the test, when a mechanically actuated testing machine is used, the beam of the testing machine shall be kept constantly in a floating position.

Cold Crushing Strength

7. The cold crushing strength in pounds per square inch shall be reported as calculated from the following formula:

$$S = \frac{W}{A}$$

where S = the cold crushing strength in pounds per square inch,
W = the total maximum load, in pounds, indicated by the testing machine, and
A = the average of the gross areas of the top and bottom of the specimen, in square inches, of the section of the specimen perpendicular to the line of application of the load.

MODULUS OF RUPTURE TEST

Apparatus

8. (*a*) Any form of standard testing machine with bearing edges as described in Paragraph (*b*) may be used for the modulus of rupture test.

NOTE.—Portable apparatus properly calibrated may be used.

(*b*) Bearing edges, 1¼ in. in diameter, shall be straight and have full contact with the specimen and they shall have a length at least equal to the width of the brick to be tested. Their bases shall be so curved or rounded as to permit free movement of the working edges. The use of bearing cylinders of the type shown in Fig. 2 is recommended, although other types may be used which will provide free movement and at the same time insure full and even bearing at all times during the test. A satisfactory alternate design is shown in Fig. 3.

Test Specimen

9. The test specimen shall be a whole 9-in. brick (9 by 4½ by 2½ in.) or its equivalent size ground or cut from a refractory shape.

Number of Tests

10. Modulus of rupture tests shall be made on at least five whole specimens, and in the case of special shapes, these shall be cut from five different shapes.

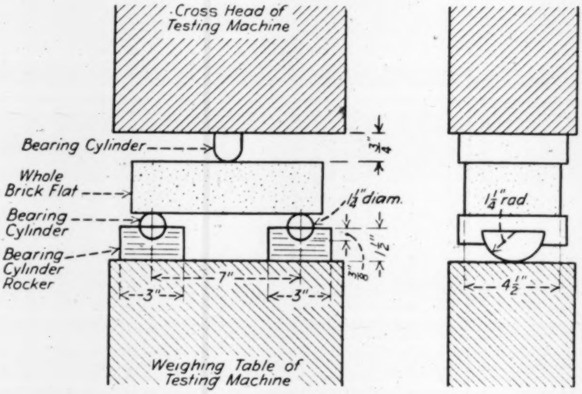
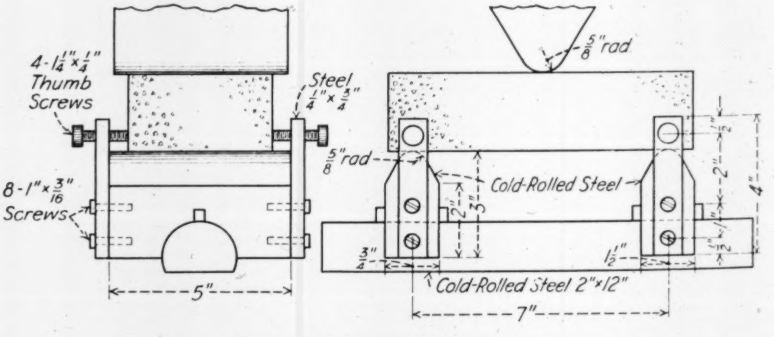

Cross Head of Testing Machine
Bearing Cylinder
Whole Brick Flat
Bearing Cylinder
Bearing Cylinder Rocker
3/4"
1 1/4" diam.
1 1/2"
3/8"
7"
3"
3"
Weighing Table of Testing Machine
1 1/4" rad.
4 1/2"

FIG. 2.—Recommended Design for Bearing Cylinders and Rockers for Use in Modulus of Rupture Test.


4-1 1/4"×1/4" Thumb Screws
Steel 1/4"×3/4"
8-1"×3/16" Screws
5"
5/8" rad
5/8" rad
Cold-Rolled Steel
3"
2"
3/4"
1 1/2"
Cold-Rolled Steel 2"×12"
7"
1/2"
2"
1"
1/2"
4"

Transverse Brick Testing Apparatus

FIG. 3.—Recommended Design for Alternate Apparatus for Modulus of Rupture Test.

Modulus of Rupture Test

11. (*a*) A test specimen shall be placed flatwise on the bearing cylinders with a span of 7 in. and the load applied at mid-span.

(*b*) The speed of head of the testing machine, during the application of the load, shall not be more than 0.05 in. per min.

Modulus of Rupture

12. The modulus of rupture in pounds per square inch shall be reported as calculated from the following formula:

$$R = \frac{3Wl}{2bd^2}$$

where R = the modulus of rupture in pounds per square inch,
W = the total load in pounds at which the specimen failed,
l = the distance between the supports in inches,
b = the width of the specimen in inches, and
d = the depth of the specimen in inches.

TENTATIVE DEFINITIONS OF TERMS RELATING TO REFRACTORIES[1]

A.S.T.M. Designation: C 71 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1936; REVISED, 1937.

Insulating Brick.—A product manufactured in standard brick shapes used primarily for heat insulation purposes.

Insulating Fire Brick.—A product with low-heat conductivity suitable for use as a lining for furnaces.

Calcining.—In the case of refractories manufacture, the heating of raw materials to redness or a higher temperature.

Burning (*Firing*).—In the case of refractories manufacture, the heat treatment of a refractory mix at a temperature high enough to develop a bond by partial vitrification.

Hard-Burned Refractory Ware.—Ware which has been subjected to a maximum intensity of heat treatment practicable.

Light-Burned Refractory Ware.—Ware which has been subjected to a minimum intensity of heat treatment practicable.

Medium-Burned Refractory Ware.—Ware which has been subjected to heat treatment of intermediate intensity.

50 per cent Alumina-Diaspore Refractories.—Refractory brick and shapes having diaspore or nodular fire clay as an essential original ingredient, an alumina content of 50 per cent ± 2.5 per cent, and a Pyrometric Cone Equivalent not lower than Standard Pyrometric Cone No. 34.

60 per cent Alumina-Diaspore Refractories.—Refractory brick and shapes having diaspore or nodular fire clay as an essential original ingredient, an alumina content of 60 per cent ± 2.5 per cent, and a Pyrometric Cone Equivalent not lower than Standard Pyrometric Cone No. 35.

70 per cent Alumina-Diaspore Refractories.—Refractory brick and shapes having diaspore as an essential original ingredient, an alumina content of 70 per cent ± 2.5 per cent, and a Pyrometric Cone Equivalent not lower than Standard Pyrometric Cone No. 36.

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[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

These definitions, when adopted as standard, will be added to the Standard Definitions of Terms Relating to Refractories (A.S.T.M. Designation: C 71 - 36), 1936 Book of A.S.T.M. Standards, Part II, p. 255.

NOTE.—Definitions of diaspore clay and nodular fire clay appear in the Standard Definitions of Terms Relating to Refractories (A.S.T.M. Designation: C 71) of the American Society for Testing Materials.[1]

Alumina content is determined in accordance with the Tentative Methods of Chemical Analysis of Refractory Materials (A.S.T.M. Designation: C 18 - 37 T) of the American Society for Testing Materials.[2]

Pyrometric Cone Equivalent is determined in accordance with the Standard Method of Test for Pyrometric Cone Equivalent of Fire Clay and Fireclay Brick (A.S.T.M. Designation: C 24) of the American Society for Testing Materials.[3]

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 255.

[2] See p. 760.

[3] 1936 Book of A.S.T.M. Standards, Part II, p. 229.

TENTATIVE SPECIFICATIONS
FOR
CONCRETE AGGREGATES[1]

A.S.T.M. Designation: C 33 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St , Philadelphia, Pa.

ISSUED, 1921; REVISED, 1923, 1926, 1928, 1930, 1931, 1936, 1937.

QUALITY OF AGGREGATES

It is recognized that for certain purposes satisfactory results may be obtained with materials not conforming to these specifications. In such cases the use of fine and coarse aggregates not conforming to these specifications may be authorized only under special provisions based upon laboratory studies of the possibility of designing a mixture of materials to be used on the job that will yield concrete equivalent in quality to the specified mixture made with material complying with these specifications in all respects.

FINE AGGREGATE

General Characteristics

1. Fine aggregate shall consist of natural sand, sand prepared from stone, blast-furnace slag, or gravel; or, subject to the approval of the engineer, other inert materials having similar characteristics.

Deleterious Substances

2. (*a*) The amount of deleterious substances shall not exceed the following limits:

	RECOMMENDED PERMISSIBLE LIMITS, PER CENT BY WEIGHT	MAXIMUM PERMISSIBLE LIMITS, PER CENT BY WEIGHT
Clay lumps, not more than..............	1	1.5
Coal and lignite........................	0.25	1
Material finer than No. 200 sieve:		
(*a*) In concrete subject to surface abrasion, not more than............	2	3
(*b*) All other classes of concrete, not more than.........................	3	5
Other deleterious substances (such as shale, alkali, mica, coated grains, soft and flaky particles).........................	as specified	as specified

NOTE.—The recommended limits should be specified on all work where it is economically practicable to obtain materials conforming thereto.

(*b*) All fine aggregate shall be free from injurious amounts of organic impurities. Aggregates subjected to the colorimetric test for organic impurities and producing a color darker than the standard shall be rejected unless they pass the mortar strength test as specified in Section 4.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

Grading

3. (*a*) Fine aggregate shall be well graded from coarse to fine and when tested by means of laboratory sieves[1] shall conform to the following requirements:

	PER CENT
Passing a ⅜-in. sieve	100
Passing a No. 4 sieve	(95) to (100)
Passing a No. 16 sieve	(45) to (80)
Passing a No. 50 sieve	(5) to (30)
Passing a No. 100 sieve	(0) to (8)

NOTE.—Figures in parentheses are suggested as limiting percentages but they may be altered within these limits to suit local conditions.

(*b*) The above gradation for fine aggregate represents the extreme limits which shall determine the suitability for use of fine aggregate from all sources of supply. The gradation of fine aggregate from any one source shall be reasonably uniform and not subject to the extreme percentages of gradation specified above. For the purpose of determining the degree of uniformity of a fine aggregate, a fineness modulus determination shall be made upon representative samples of fine aggregate, from such sources as are proposed for use. Fine aggregate from any one source having a variation in fineness modulus greater than 0.20 either way from the fineness modulus of the representative sample submitted by the contractor shall either be rejected or may be accepted subject to such adjustment in proportions as may be necessary by reason of changes in grading of fine aggregate. Fine aggregate from different sources of supply shall not be mixed or stored in the same pile nor used alternately in the same class of construction or mix, without permission from the engineer.

(*c*) In case the concrete resulting from a mixture of aggregates approaching the extreme limits for gradation is not of a workable character, or when finished does not exhibit a proper surface, due to an excess of particles approximately ⅛ to ½ in. in size, either a fine aggregate having a sufficiently greater percentage of fine material, or a coarse aggregate having a sufficiently smaller percentage of fine material shall be used.

Mortar Strength

4. Fine aggregate shall be of such quality that when made into a mortar and subjected to the mortar strength test (Section 10 (*e*))

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[1] For detail requirements for these sieves, see the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials, 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

it shall develop a compressive strength at 7 and 28 days of not less than .... per cent[1] of that developed by a mortar prepared in the same manner with the same cement and graded Ottawa sand having a fineness modulus of 2.40 ± 0.10.

NOTE.—The graded Ottawa sand mentioned above may be obtained with a mixture of approximately equal parts by weight of standard Ottawa sand and run-of-mine Ottawa sand.

COARSE AGGREGATE

General Characteristics

5. Coarse aggregate shall consist of crushed stone, gravel, blast-furnace slag, or other approved inert materials of similar characteristics, or combinations thereof, having hard, strong, durable pieces, free from adherent coatings and conforming to the requirements of these specifications.

Deleterious Substances

6. The amount of deleterious substances shall not exceed the following limits:

	RECOMMENDED PERMISSIBLE LIMITS, PER CENT BY WEIGHT	MAXIMUM PERMISSIBLE LIMITS, PER CENT BY WEIGHT
Soft fragments........................	2	5
Coal and lignite........................	0.25	1
Clay lumps...........................	0.25	0.25
Material finer than No. 200 sieve.........	0.5 (Note 2)	1 (Note 2)
Other deleterious substances.............	as specified	as specified

NOTE 1.—The recommended requirements should be specified on all work where it is economically practicable to obtain materials conforming thereto.

NOTE 2.—When the material finer than the No. 200 sieve consists essentially of crusher dust the recommended and maximum permissible limits specified above may be raised to 0.75 and 1.5 per cent, respectively.

Grading

7. (*a*) Coarse aggregate shall be well graded, between the limits specified, and shall conform to the following requirements:

CRUSHED STONE, GRAVEL AND BLAST-FURNACE SLAG

Designated Size	Percentages Passing Laboratory Sieves Having Square Openings						
	2 in.	1½ in.	1 in.	¾ in.	½ in.	⅜ in.	No. 4
2 in. to No. 4..................	95 to 100	....	35 to 70	....	10 to 30	....	0 to 5
1½ in. to No. 4.................	....	95 to 100	....	35 to 70	....	10 to 30	0 to 5
1 in. to No. 4..................	....	....	90 to 100	....	25 to 60	....	0 to 10
¾ in. to No. 4..................	....	....	....	90 to 100	....	20 to 55	0 to 10
½ in. to No. 4[a]................	....	....	....	....	90 to 100	....	0 to 15
2 in. to 1 in....................	90 to 100	35 to 70	0 to 15	....	....	....	......
1½ in. to ¾ in..................	....	90 to 100	20 to 55	0 to 15	....	....	......

[a] Not more than 5 per cent passing the No. 8 sieve.

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[1] A percentage should be inserted by the engineer to suit local conditions.

(*b*) Designation of a given coarse aggregate as aggregate of a certain maximum size shall be understood to mean that more than 5 per cent must be retained on the next smaller of the sieves appearing in the above table.

(*c*) In case the concrete resulting from a mixture of aggregates approaching the extreme limits for gradation is not of a workable character, or when finished does not exhibit a proper surface, due to an excess of particles approximately $\frac{1}{8}$ to $\frac{1}{2}$ in. in size, either a fine aggregate having a sufficiently greater percentage of fine material or a coarse aggregate having a sufficiently smaller percentage of fine material shall be used.

Weight of Slag

8. Blast-furnace slag that meets the grading requirements of these specifications shall conform to the following minimum weight requirements:

General concrete	65 lb. per cu. ft.
Concrete subject to abrasion	70 lb. per cu. ft.

Durability

9. Coarse aggregate shall pass a sodium sulfate accelerated soundness test, except that aggregates failing in the accelerated soundness test may be used if they pass a satisfactory freezing and thawing test.

NOTE.—Many engineers believe that an abrasion test for coarse aggregate to be used in concrete subject to abrasion is important, but no test limits are specified, due to the status of knowledge concerning suitable specification limits for this test. The committee believes that the abrasion tests when applied to blast-furnace slag do not meet the requirements for a desirable test. This recommendation is made after consideration of the results of a study to determine the value of the abrasion test as to the concrete making properties of slag.

METHODS OF SAMPLING AND TESTING

Methods of Testing

10. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials, except as specified in Paragraphs (*j*) and (*k*):

(*a*) *Sampling.*—Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (A.S.T.M. Designation: D 75).[1]

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1092.

(*b*) *Sieve Analysis.*—Standard Method of Test for Sieve Analysis of Aggregates for Concrete (A.S.T.M. Designation: C 41).[1]

(*c*) *Amount of Material Finer than No. 200 Sieve.*—Standard Method of Test for Amount of Material Finer than No. 200 Sieve in Aggregates (A.S.T.M. Designation: C 117).[2]

(*d*) *Organic Impurities.*—Standard Method of Test for Organic Impurities in Sands for Concrete (A.S.T.M. Designation: C 40).[3]

(*e*) *Mortar Strength.*—Standard Method of Test for Structural Strength of Fine Aggregate Using Constant Water-Cement-Ratio Mortar (A.S.T.M. Designation: C 87).[4]

(*f*) *Compressive Strength.*—Standard Methods of Making Compression Tests of Concrete (A.S.T.M. Designation: C 39).[5]

(*g*) *Soundness.*—Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (A.S.T.M. Designation: C 88 - 37 T).[6]

(*h*) *Freezing and Thawing.*—Method of freezing-and-thawing tests of drain tile as described in the Standard Specifications for Drain Tile (A.S.T.M. Designation: C 4).[7]

(*i*) *Coal and Lignite.*—Tentative Method of Test for Coal and Lignite in Sand (A.S.T.M. Designation: C 123 - 36 T).[8]

(*j*) *Shale.*—Method of Test for Percentage of Shale in Aggregate,[9] Tentative Method T-10 of The American Association of State Highway Officials.

(*k*) *Soft Fragments.*—Method of Test for Quantity of Soft Pebbles in Gravel,[9] Tentative Method T-8 of The American Association of State Highway Officials.

(*l*) *Moisture.*—Standard Method of Test for Surface Moisture in Fine Aggregate (A.S.T.M. Designation: C 70).[10]

(*m*) *Weight of Slag.*—Standard Method of Test for Unit Weight of Aggregate for Concrete (A.S.T.M. Designation: C 29).[11]

(*n*) *Abrasion.*—If abrasion tests are made the following methods of test are recommended:

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 353.

[2] 1937 Supplement to Book of A.S.T.M. Standards, p. 99.

[3] 1936 Book of A.S.T.M. Standards, Part II, p. 350.

[4] *Ibid.*, p. 355.

[5] *Ibid.*, p. 342.

[6] See p. 804.

[7] 1936 Book of A.S.T.M. Standards, Part II, p. 257.

[8] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 794 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 593.

[9] See "Tentative Standard Specifications for Highway Materials and Methods of Sampling and Testing," The Am. Assn. State Highway Officials, pp. 98, 97 (1935), also 1928 Report of Committee C-9 on Concrete and Concrete Aggregates, see *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part I, pp. 362, 363 (1928).

[10] 1936 Book of A.S.T.M. Standards, Part II, p. 357.

[11] *Ibid.*, p. 364.

(1) *Abrasion of Gravel.*—Tentative Method of Test for Abrasion of Gravel by Use of the Deval Machine (A.S.T.M. Designation: D 289 - 37 T).[1]

(2) *Abrasion of Rock.*—Standard Method of Test for Abrasion of Rock by Use of the Deval Machine (A.S.T.M. Designation: D 2),[2] except that for specific gravities lower than 2.2 a 4000-g. sample shall be used.

(*o*) *Fineness Modulus.*—The fineness modulus is the sum of percentages in the sieve analysis divided by 100 when the sieve analysis is expressed as cumulative percentages coarser than each of sieves Nos. 100, 50, 30, 16, 8, 4, etc. The sieves shall conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3] The sieve analysis shall be made in accordance with the Standard Method of Test for Sieve Analysis of Aggregates for Concrete (A.S.T.M. Designation: C 41) of the American Society for Testing Materials.[4]

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[1] See p. 921.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 1040.

[3] *Ibid.*, p. 1413.

[4] *Ibid.*, p. 353.

TENTATIVE SPECIFICATIONS
FOR
LIGHTWEIGHT AGGREGATE FOR CONCRETE[1]

A.S.T.M. Designation: C 130 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover lightweight aggregates for use in concrete.

NOTE.—The specification limits which are not enclosed in parenthesis are recommended for use when it is practicable to secure economically materials conforming to them. When it is not practicable to secure such materials economically the specification limits may be extended but not beyond the maximum permissible limits shown in parentheses. Generally, such extension in the specification limits can be compensated for by a change in the design of the mix or by the exercise of more than the usual care in the control of the concrete during construction.

General Characteristics

2. (*a*) Lightweight aggregates shall consist of pumice, lava, tufa, slag, burned clay, burned shale, cinders derived from the high-temperature combustion of coal or coke showing a loss on ignition of not more than 25 per cent (40 per cent)[2] and volatile matter of not more than 5 per cent, or, subject to approval by the engineer, other material having strong, durable particles and conforming to the requirements of these specifications.

(*b*) When approved by the engineer, for the purpose of securing the desired workability, lightweight aggregate meeting the requirements of these specifications may be mixed with a fine aggregate meeting the requirements of the Tentative Specifications for Concrete Aggregates (A.S.T.M. Designation: C 33 – 37 T) of the American Society for Testing Materials,[3] provided, however, that the combined aggregate shall meet all the other requirements of these specifications.

Grading

3. Lightweight aggregate shall be graded from coarse to fine within the following limits:

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] See Note under Section 1.

[3] See p. 792.

Size Designation	Percentages Passing Sieves Having Square Openings								
	1 in.	¾ in.	½ in.	⅜ in.	No. 4	No. 8	No. 16	No. 50	No. 100
Fine Aggregate:									
¼ in. to dust	....	....	....	100	95 to 100	....	45 to 80	10 to 30	5 to 15
⅜ in. to dust	....	....	100	95 to 100	55 to 80	....	....	10 to 25	5 to 15
Coarse Aggregate:									
½ in. to No. 4	....	100	90 to 100	40 to 75	0 to 15	0 to 5	....	....	....
¾ in. to No. 4	100	90 to 100	....	20 to 55	0 to 10	0 to 5	....	....	....

Unit Weight

4. Lightweight aggregate meeting the grading requirements of these specifications when tested in a dry condition shall conform to the following weight requirements:

	UNIT WEIGHT, LB. PER CU. FT.
Fine aggregate, not more than	70
Coarse aggregate, not more than	55[a]

[a] Heavier aggregate may be permitted when lightweight aggregates meeting the above requirements are not economically available and the higher weight of concrete resulting from their use is permissible.

Mortar Strength

5. (*a*) Lightweight aggregate shall be of such quality that when made into a mortar and subjected to the mortar strength test (Section 9 (*d*)) it shall develop a compressive strength at 7 or 28 days of not less than 70 per cent (or, for concrete not exposed to wear or weather, 60 per cent), of that developed by a mortar prepared in the same manner, with the same cement and the graded Ottawa sand described in Paragraph (*b*). Coarse aggregate shall be reduced to the grading specified for fine aggregate before use in the mortar test.

(*b*) The Ottawa sand referred to in Paragraph (*a*) is natural silica sand produced by the Ottawa Silica Co., Ottawa, Ill., graded as follows:[1]

Retained on No. 100 sieve	98 ± 2 per cent
Retained on No. 50 sieve	72 ± 5 per cent
Retained on No. 30 sieve	2 ± 2 per cent
Retained on No. 16 sieve	0 per cent

Soundness

6. Lightweight aggregate, when subjected to five cycles of the accelerated sulfate soundness test, shall lose not more than 12 per cent (15 per cent)[2] in weight, provided, however, that an aggregate failing in this requirement may be accepted if it passes a satisfactory freezing-and-thawing test. The engineer may waive the soundness test requirement for lightweight aggregate for concrete not to be exposed to moisture.

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[1] These requirements for Ottawa sand are identical with those specified in the Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 - 37 T), see p. 726.

[2] See Note under Section 1.

Deleterious Substances

7. Lightweight aggregate shall not contain excessive amounts of deleterious substances.

Organic Impurities

8. All lightweight aggregate shall be free from injurious amounts of organic impurities. Aggregates subjected to the colorimetric test for organic impurities and producing a color darker than the standard shall be rejected unless they pass the mortar strength test (Section 5), which requirement may be raised by the engineer by not more than 10 per cent.

Methods of Sampling and Testing

9. Lightweight aggregate shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Standard Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (A.S.T.M. Designation: D 75).[1]

(*b*) *Grading.*—Standard Method of Test for Sieve Analysis of Aggregates for Concrete (A.S.T.M. Designation: C 41).[2]

(*c*) *Unit Weight.*—Standard Method of Test for Unit Weight of Aggregate for Concrete (A.S.T.M. Designation: C 29),[3] except that shaking to refusal for each layer shall be substituted for tamping as specified. Unit weights shall be determined on material dried to constant weights at temperatures of not less than 100 C. and not more than 120 C.

(*d*) *Mortar Strength.*—Standard Method of Test for Structural Strength of Fine Aggregate Using Constant Water-Cement-Ratio Mortar (A.S.T.M. Designation: C 87).[4]

(*e*) *Compressive Strength.*—Standard Methods of Making Compression Tests of Concrete (A.S.T.M. Designation: C 39).[5]

(*f*) *Soundness.*—Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (A.S.T.M. Designation: C 88 - 37 T).[6]

(*g*) *Organic Impurities.*—Standard Method of Test for Organic Impurities in Sands for Concrete (A.S.T.M. Designation: C 40).[7]

(*h*) *Freezing and Thawing.*—Method of freezing-and-thawing test as described in Sections 26 to 31 of the Standard Specifications for Drain Tile (A.S.T.M. Designation: C 4).[8]

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1092.
[2] *Ibid.*, p. 353.
[3] *Ibid.*, p. 364.
[4] *Ibid.*, p. 355.
[5] *Ibid.*, p. 342.
[6] See p. 804.
[7] 1936 Book of A.S.T.M. Standards, Part II, p. 350.
[8] *Ibid.*, p. 257.

TENTATIVE METHOD OF TEST
FOR
ABRASION OF COARSE AGGREGATE BY USE OF THE LOS ANGELES MACHINE[1]

A.S.T.M. Designation: C 131 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. This method of test is intended for determining the abrasive resistance of crushed rock, crushed slag, uncrushed gravel, and crushed gravel (Note).

NOTE.—Ledge rock, hand-broken into approximately cubical fragments of the different sizes shown, when tested by this method, has been found to have a loss of approximately 85 per cent of that for crushed rock of the same quality.

Los Angeles Machine

2. The Los Angeles abrasion testing machine shall be used. The machine shall consist of a hollow iron drum, having inside dimensions of 20 in. in length and 28 in. in diameter, rotating on a horizontal axis. An opening in the cylinder shall be provided for the introduction of the sample and shall be closed dust-tight, with a removable cover bolted into place. A shelf, which projects $3\frac{1}{2}$ in. into the drum and extends the full length of the drum, shall be attached to the cover or to the inside of the drum. The surface of the shelf which catches the charge shall be rectangular and shall lie in a radial plane. The testing machine shall conform in all of its essential characteristics to the plan shown in Fig. 1.

Abrasive Charge

3. (*a*) The abrasive charge shall consist of cast-iron spheres or steel spheres approximately $1\frac{7}{8}$ in. in diameter and weighing between 405 and 440 g. each. The cast-iron spheres shall conform to the following requirements as to chemical composition:[2]

Combined carbon, min., per cent	2.50
Graphitic carbon, max., per cent	0.25
Silicon, max., per cent	1.00
Manganese, max., per cent	0.50
Phosphorus, max., per cent	0.25
Sulfur, max., per cent	0.08

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[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates and Committee D-4 on Road and Paving Materials.

[2] These requirements are identical with those for the cast-iron spheres used in the rattler test for paving brick as prescribed in the Tentative Specifications for Paving Brick (A.S.T.M. Designation: C 7 - 37 T) of the American Society for Testing Materials, p. 740.

(*b*) A charge of 12 spheres weighing 5000 g. ± 25 g. shall be used with the grading *A* described in Section 4, and a charge of 11 spheres weighing 4583 g. ± 25 g. shall be used with the grading *B* described in Section 4.

Test Sample

4. The test sample shall consist of 5000 g. of clean, dry aggregate and shall conform to either of the following gradings. The grading used shall be that most nearly representing the aggregate furnished for the work.

SIEVE SIZE (SQUARE OPENINGS) PASSING	RETAINED ON	GRADING *A* WEIGHT, G.	GRADING *B* WEIGHT, G.
1½ in.	1 in.	1250	0
1 in.	¾ in.	1250	0
¾ in.	½ in.	1250	2500
½ in.	⅜ in.	1250	2500

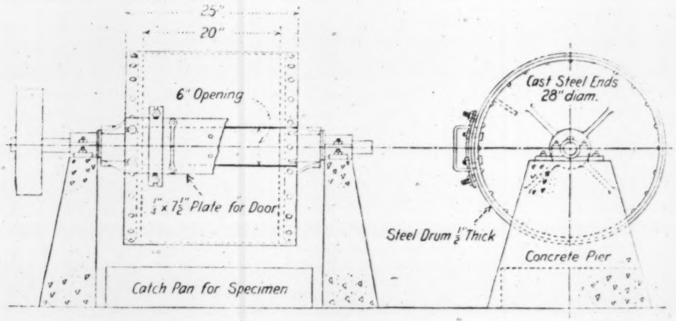

FIG. 1.—Los Angeles Abrasion Machine.

Procedure

5. The test sample and the abrasive charge shall be placed in the Los Angeles abrasion testing machine and the machine rotated for 500 revolutions at a speed of from 30 to 33 r.p.m. If an angle is used as the shelf, the machine shall be rotated in such a direction that the charge is caught on the outside surface of the angle. At the completion of the test, the material shall be removed from the machine and sieved on a No. 12 sieve conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[1] The material retained on the sieve shall be washed, dried, and accurately weighed to the nearest gram (Note).

NOTE.—Attention is called to the fact that valuable information concerning the uniformity of the sample under test may be obtained by determining the loss after 100 revolutions; when this determination is made care should be taken to avoid loss of any part of the sample; the entire sample, including the dust of abrasion, shall be returned to the testing machine for the completion of the test.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

Calculation

6. The difference between the original weight and the final weight of the test sample shall be expressed as a percentage of the original weight of the test sample. This value shall be reported as the percentage of wear.

TENTATIVE METHOD OF TEST

FOR

SOUNDNESS OF AGGREGATES BY USE OF SODIUM SULFATE OR MAGNESIUM SULFATE[1]

A.S.T.M. Designation: C 88 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to **annual revision.** Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1931; Revised, 1932, 1935, 1937.

Scope

1. This method describes the procedure to be followed in testing aggregates to determine their resistance to disintegration by saturated solutions of sodium sulfate or magnesium sulfate. It furnishes information helpful in judging of the soundness of aggregates subjected to weathering action. However, in selecting aggregates, principal dependence should be placed on service records of the materials when exposed to actual weathering conditions. In the absence of such information, the test method affords a useful guide. Because of the limited amount of information concerning the significance of the test results and because of the lack of uniformity of tests made in different laboratories and of repeated tests in the same laboratory, the test method should not be used as an arbitrary basis of rejection; it should be used only to furnish information to indicate whether or not the materials require further investigation of their soundness.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Solution.*—Supply of saturated solution of sodium sulfate or magnesium sulfate prepared in accordance with Section 3 (*a*) and (*b*).

(*b*) *Sieves.*—Sieves, sizes Nos. 100, 50, 30, 16, 8 and 4, conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials,[2] and sieves with square openings in sizes $\frac{3}{8}$ in., $\frac{3}{4}$ in., $1\frac{1}{2}$ in., $2\frac{1}{2}$ in., and larger sizes by 1-in. spread for screening the samples in accordance with Sections 4 and 5.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

This method is in effect a combination, without change in principle, of the Tentative Methods of Test for Soundness of Fine Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 - 35 T) and Test for Soundness of Coarse Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 89 - 35 T); see *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, pp. 842, 837 (1935); also 1936 Book of A.S.T.M. Tentative Standards, pp. 507, 502.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

(*c*) *Containers.*—Suitable container (Note) for immersing the samples of aggregate in the solution in accordance with the procedure hereinafter described shall be selected. Containers shall be non-metallic or of metal not subject to corrosion by sodium sulfate or magnesium sulfate.

NOTE.—No standard form of container is recommended since different practices will be most convenient in various laboratories. The committee has had presented to it data indicating that the type of container may affect the results in some degree, but these data are not sufficiently comprehensive to permit of specifying a particular type. Two general methods of immersion have been used as follows:

(*1*) Each sample placed in a water-tight container and covered with the sodium sulfate or magnesium sulfate solution.

(*2*) Each sample placed in a container with a wire mesh or perforated bottom (depending upon size of particles being tested) and immersed in a vat containing the sodium sulfate or magnesium sulfate solution. Care should be taken to prevent loss by flotation if uncovered containers are submerged. If nested sieves are used, a pan should be inserted between sieves to prevent loss of material from one sieve to another.

(*d*) *Temperature Regulation.*—Suitable means for regulating the temperature of the samples during immersion in the sodium sulfate or magnesium sulfate solution shall be provided.

(*e*) *Balances.*—For fine aggregate a balance having a capacity not less than 500 g., sensitive to at least 0.1 g., shall be used; for coarse aggregate a balance having a capacity not less than 5000 g., sensitive to at least 1 g. shall be used.

(*f*) *Drying Oven.*—The drying oven shall provide a free circulation of air through the oven and shall be capable of maintaining a temperature of 105 to 110 C. (221 to 230 F.).

Sodium Sulfate Solution

3. (*a*) The saturated solution of sodium sulfate shall be prepared by dissolving a c.p., U.S.P., or equal grade of salt in water at a temperature of 25 to 30 C. (77 to 84 F.). Sufficient salt (Note), either of the anhydrous (Na_2SO_4) or of the crystalline ($Na_2SO_4 \cdot 10H_2O$) form, shall be added to insure not only saturation but also the presence of excess crystals when the solution is ready for use in the tests. The mixture shall be thoroughly stirred during the addition of the salt and the solution shall be stirred at frequent intervals until used. The solution shall be cooled to a temperature of 21 C. ± 1 C. (70 F. ± 2 F.) and maintained at that temperature for at least 48 hr before use; it shall be thoroughly stirred immediately before use and when used shall have a specific gravity of not less than 1.151 and not greater than 1.174.

NOTE.—For the solution, 215 g. of anhydrous salt or 485 g. of the decahydrate per liter of water are sufficient for saturation at 22 C. However, since these salts are not completely stable and since it is desirable that an excess of crystals be present, the use of not less than 350 g. of the anhydrous salt or 500 g. of the hydrous salt per liter of water is recommended.

Magnesium Sulfate Solution

(*b*) The saturated solution of magnesium sulfate shall be made by dissolving a c.p., U. S. P., or equal grade of salt in water at a temperature of

25 to 30 C. (77 to 84 F.). Sufficient salt (Note), either of the anhydrous ($MgSO_4$) or of the crystalline ($MgSO_4 \cdot 7H_2O$) (Epsom Salt) form, shall be added to insure saturation and the presence of excess crystals when the solution is ready for use in the tests. The mixture shall be thoroughly stirred during the addition of the salt and the solution shall be stirred at frequent intervals until used. The solution shall be cooled to a temperature of 21 C. ± 2 C. (70 F. ± 4 F.) and maintained at that temperature for at least 48 hr. before use; it shall be thoroughly stirred immediately before use and when used shall have a specific gravity of not less than 1.295 and not greater than 1.308.

NOTE.—For the solution, 350 g. of anhydrous salt or 725 g. of the heptahydrate per liter of water are sufficient for saturation at 23 C. However, since these salts are not completely stable, with the hydrous salt being the more stable of the two, and since it is desirable that an excess of crystals be present, it is recommended that the hydrous salt be used and in an amount of not less than 800 g. per liter of water.

Samples

4. (*a*) *Fine Aggregate.*—Fine aggregate for the test shall be passed through a ⅜-in. sieve. The sample shall be of such size that it will yield not less than 100 g. of each of the following sizes, which are available in amounts of 5 per cent or more, expressed in terms of the following A.S.T.M. sieves:

Retained on sieve No. 100, passing sieve No. 50
Retained on sieve No. 50, passing sieve No. 30
Retained on sieve No. 30, passing sieve No. 16
Retained on sieve No. 16, passing sieve No. 8
Retained on sieve No. 8, passing sieve No. 4
Retained on sieve No. 4, passing ⅜-in. sieve

(*b*) *Coarse Aggregate.*—Coarse aggregate for the test shall consist of material from which the sizes finer than the No. 4 sieve have been removed. The sample shall be of such a size that it will yield not less than the following amounts of the different sizes, which are available in amounts of 5 per cent or more:

No. 4 to ⅜-in. size (square openings)	100 g.
⅜ to ¾-in. size (square openings)	300 g.
¾ to 1½-in. size (square openings)	1500 g.
1½ to 2½-in. size (square openings)	3000 g.
Larger sizes by 1-in. spread in sieve size, each fraction	3000 g.

(*c*) Should the samples contain less than 5 per cent of any of the sizes specified in Paragraph (*a*) or (*b*), that size shall not be tested, but, for the purpose of calculating the test results, it shall be considered to have the same loss in sodium sulfate or magnesium sulfate treatment as the average of the next smaller and the next larger size, or if one of these sizes is absent, it shall be considered to have the same loss as the next larger or next smaller size, whichever is present.

Preparation of Test Sample

5. (*a*) *Fine Aggregate.*—The sample of fine aggregate shall be thoroughly washed on a No. 100 sieve and dried to constant weight at a temperature of 105 to 110 C. (221 to 230 F.) and separated into the different sizes by sieving, as follows:

Make a rough separation of the graded sample by means of a nest of the standard sieves specified in Section 4 (*a*). From the fractions obtained in this manner select samples of sufficient size to yield 100 g. after sieving to refusal. (In general, a 110-g. sample will be sufficient.) Fine aggregate sticking in the meshes of the sieves shall not be used in preparing the samples. Samples consisting of 100 g. shall be weighed out of each of the separated fractions after final sieving and placed in separate containers for the test.

(*b*) *Coarse Aggregate.*—The sample of coarse aggregate shall be thoroughly washed and dried to constant weight at a temperature of 105 to 110 C. (221 to 230 F.) and shall be separated into the different sizes shown in Section 4 (*b*) by screening to refusal. The proper weight of sample for each fraction shall be weighed out and placed in separate containers for the test. In the case of fractions coarser than the $\frac{3}{4}$-in. sieve, the number of particles shall be counted.

(*c*) *Ledge Rock.*—For testing ledge rock, the sample shall be prepared by breaking it into fragments reasonably uniform in size and shape and weighing approximately 100 g. each. The test sample shall weigh 5000 g. ±2 per cent. The sample shall be thoroughly washed and dried previous to test as described in Paragraph (*b*).

Storage of Samples in Solution

6. The samples shall be immersed in the prepared solution of sodium sulfate or magnesium sulfate for 18 hr. in such a manner that the solution covers them to a depth of at least $\frac{1}{2}$ in. The containers shall be covered to reduce evaporation and prevent the accidental addition of extraneous substances. The samples immersed in the solution shall be maintained at a temperature of 21 C. ± 1 C. (70 F. ± 2 F.) for the 18-hr. period.

Drying Samples After Immersion

7. After the 18-hr. immersion period the aggregate samples shall be removed from the solution and placed in the drying oven. The temperature of the oven shall have been brought previously to 105 to 110 C. (221 to 230 F.). Care shall be exercised to avoid loss of any of the aggregate particles or, in the case of fine aggregate, of any detritus coarser than a No. 100 sieve (Note). The samples shall be dried to constant weight at the specified temperature. After drying, the samples shall be allowed to cool to room temperature, when they shall again be immersed in the prepared solution as described in Section 6.

NOTE.—In the case of coarse aggregate the detritus should also be saved if the complete analysis suggested in the Note in Section 9 (*b*) is made.

Cycles

8. The process of alternate immersion and drying shall be repeated until the required number of cycles is obtained.

Quantitative Examination

9. The quantitative examination (Note) shall be made as follows:

(*a*) After the completion of the final cycle, and after the sample is cooled the sample shall be washed free from the sodium sulfate or magnesium sulfate as determined by the reaction of the wash water with barium chloride ($BaCl_2$).

(*b*) After the sodium sulfate or magnesium sulfate solution has been removed, each fraction of the sample shall be dried to constant weight at 105 to 110 C. (221 to 230 F.); weighed, and, except in the case of ledge rock, screened over the same sieve on which it was retained before the test. The particles retained on this sieve shall be weighed and the weight recorded.

NOTE.—In addition to the procedure described in Paragraphs (*a*) and (*b*), it is suggested that additional information of value will be obtained by examining each fraction visually in order to determine if there is any evidence of excessive splitting of the grains. It is suggested also that additional information of value will be obtained if, after treating each separate fraction of the sample as described in Paragraph (*b*), all sizes, including detritus, shall be combined and a sieve analysis made using a complete set of sieves for the determination of the fineness modulus. The results of the sieve analysis shall be recorded as cumulative percentages retained on each sieve.

(*c*) In the case of ledge rock the loss in weight shall be determined by subtracting from the original weight of the sample the final weight of all fragments which have not broken into three or more pieces.

Qualitative Examination

10. (*a*) Fractions of samples coarser than $\frac{3}{4}$ in. shall be examined qualitatively after each immersion and quantitatively at the completion of the test.

(*b*) The qualitative examination and record shall consist of two parts: (*1*) Observing the effect of the action (Note) by the sodium sulfate or magnesium sulfate solution and the nature of the action, and (*2*) Counting the number of particles affected.

NOTE.—Many types of action may be expected. In general, they may be classified as disintegration, splitting, crumbling, cracking, flaking, etc.

While only particles larger than the $\frac{3}{4}$ in. in size are required to be examined qualitatively, it is recommended that examination of the smaller sizes be made in order to determine if there is any evidence of excessive splitting.

Report

11. The report shall include the following data (Note):

(*a*) Weight of each fraction of each sample before test.

(*b*) Except in the case of ledge rock, material from each fraction of the sample, finer than sieve on which fraction was retained before test, expressed as a percentage by weight of the fraction.

(*c*) Weighted average calculated from the percentage of loss for each fraction, based on the grading of the sample as received for examination, or preferably, on the average grading of the material from that portion of the supply of which the sample is representative.

(*d*) In the case of particles coarser than $\frac{3}{4}$ in. before test:

(*1*) Number of particles in each fraction before test, and

(*2*) Number of particles affected, classified as to number disintegrating, splitting, crumbling, cracking, flaking, etc.

(*e*) In the case of ledge rock:

(*1*) The percentage of loss calculated as described in Section 9 (*c*) and

(*2*) The number of pieces affected, classified as to number disintegrating, splitting, crumbling, cracking, flaking, etc.

(*f*) Character of solution (sodium or magnesium sulfate).

NOTE.—The following table, shown with test values inserted for purpose of illustration, is a suggested form for recording test data. The test values shown might be appropriate for either salt, depending on the quality of the aggregate.

Sieve Size		Grading of Original Sample, per cent	Weight of Test Fractions Before Test, g.	Percentage Passing Finer Sieve After Test (Actual per cent Loss)	Weighted Average (Corrected per cent Loss)
Passing	Retained on				
SOUNDNESS TEST OF FINE AGGREGATE					
No. 100		5.0	....	....	....
No. 50	No. 100	11.4	100	3.5	0.40
No. 30	No. 50	26.0	100	4.2	1.09
No. 16	No. 30	25.2	100	4.8	1.21
No. 8	No 16	17.0	100	8.0	1.36
No. 4	No. 8	10.8	100	11.2	1.21
$\frac{3}{8}$ in.	No. 4	4.6	....	11.2[a]	0.52
Totals		100.0	500	....	5.79
SOUNDNESS TEST OF COARSE AGGREGATE					
$2\frac{1}{2}$ in.	$1\frac{1}{2}$ in.	20.0	3000[b]	4.8	0.96
$1\frac{1}{2}$ in.	$\frac{3}{4}$ in.	45.0	1500[b]	8.0	3.60
$\frac{3}{4}$ in.	$\frac{3}{8}$ in.	23.0	300[b]	9.6	2.20
$\frac{3}{8}$ in.	No. 4	12.0	100[b]	11.2	1.34
Totals		100.0	4900	....	8.10

[a] The percentage loss (11.2 per cent) of the next smaller size is used as the percentage loss for this size, since this size contains less than 5 per cent of the original sample as received. See Section 4 (*c*).

[b] Minimum amounts; larger samples may be used.

TENTATIVE METHOD OF TEST
FOR
GRINDABILITY OF COAL BY THE BALL-MILL METHOD[1]

A.S.T.M. Designation: D 408 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1935; Revised, 1937.

Scope

1. This method describes a laboratory procedure for estimating the grindability of coal.[2] Briefly, the relative amounts of energy necessary to pulverize different coals is determined by placing a sample of coal in a ball mill and finding the number of revolutions required to grind it so that 80 per cent of the sample passes a 74-micron (No. 200) A.S.T.M. sieve.

Apparatus

2. The apparatus for the grindability test shall consist of the following:

(*a*) *Ball Mill.*—A cylindrical steel mill 8 in. in length and $8\frac{1}{8}$ in. in diameter, inside dimensions, and $\frac{1}{4}$ in. in thickness shall be used. Three steel lifters, each $\frac{7}{16}$ in. square and 8 in. in length, shall be riveted longitudinally at 120-deg. intervals to the inside wall of the mill. They shall be so fastened that no space is left between the wall and the lifters. The lid shall be fitted to the mill by means of a rubber gasket and held securely in place by the yoke and crossbar arrangement ordinarily employed with laboratory porcelain jar mills. A suitable mill can be made of a section of ordinary 8-in. water pipe with a bottom and lid of $\frac{1}{4}$-in. steel plate. The bottom may be welded to the pipe section.

(*b*) *Ball Charge.*—The charge for the mill shall consist of 100 steel ball bearings, each 1 in. in diameter.

Note.—Ball bearings are specified because they are of uniform diameters and smoothly polished.

(*c*) *Rotation of Mill.*—The mill shall be rotated in a horizontal position about its cylindrical axis at 40 r.p.m. The jar-mill frame used for pulverizing coal samples for analysis is suitable for this purpose. A revolution-counter shall be used to show the number of revolutions of the mill.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke.

[2] For information concerning the experimental work on which this method is based, see the paper by H. F. Yancey, O. L. Furse and R. A. Blackburn, "Estimation of the Grindability of Coal," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 108, p. 267 (1934); for further work, see C. G. Black, "Investigation of Procedure for Determination of Coal Grindability by the Ball-Mill Method," and H. F. Yancey and M. R. Geer, "Further Investigation of Methods for Estimating the Grindability of Coal," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 119, pp. 330 and 353 (1936), respectively.

(*d*) *Sieves.*—The following sieves will be required: 1680-micron (No. 12), 8 in. or more in diameter; and 74-micron (No. 200), 8 in. in diameter for machine sieving and 10 in. in diameter for hand sieving. The sieves shall conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[1]

Gross Sample

3. A representative gross sample of coal shall be collected and prepared in accordance with the Standard Method of Sampling Coal (A.S.T.M. Designation: D 21) of the American Society for Testing Materials.[2] Approximately 10 lb. of coal crushed to pass a 4760-micron (No. 4) sieve shall constitute the laboratory sample.

Preparation of Sample

4. (*a*) The 10-lb. laboratory sample shall be stage-crushed by smooth-faced double rolls[3] or other suitable crusher[4] (Paragraph (*b*)) in such a manner as to minimize the production of fine sizes. If rolls are used, the 10-lb. sample shall be passed in a thin stream through a laboratory-type double-roll crusher set with a spacing of $\frac{1}{4}$ in. between the rolls. The sample shall then be divided into two equal parts by riffling and one part discarded. The remaining 5-lb. portion shall then be screened on the 1680-micron (No. 12) sieve and the oversize passed through the crusher with the rolls set $\frac{3}{16}$ in. apart. The crushed product shall be again screened on the 1680-micron (No. 12) sieve and the undersize combined with that previously obtained. The oversize now remaining on the 1680-micron (No. 12) sieve shall be reduced in size by passing it successively through the crusher with the clearance between the rolls set at $\frac{1}{8}$, $\frac{1}{16}$, and $\frac{1}{32}$ in., respectively. Material finer than the 1680-micron (No. 12) sieve shall be removed after each crushing and only the oversize shall be crushed to the next closer roll setting. The 5-lb. lot shall then be thoroughly mixed and divided by riffling into two equal parts, one of which shall be set aside as a reserve sample. The other part shall be air-dried at room temperature to constant weight and then screened on the 74-micron (No. 200) sieve to remove the undersize, which shall be discarded. Usually, this undersize product should not exceed 5 per cent.

(*b*) If other types of laboratory crushers are used, the 10-lb. laboratory sample shall be treated as described in Paragraph (*a*) except as to the type of crusher.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

[2] *Ibid.*, p. 382.

[3] C. E. Baltzer and H. P. Hudson, "A Method for Rating the Grindability or Pulverizability of Coal, Developed by the Fuel Research Laboratories (F.R.L.), Department of Mines, Canada," *Report No. 737-1*, Department of Mines, Mines Branch, Canada (1933).

[4] C. G. Black, "Investigation of Procedure for Determination of Coal Grindability by the Ball-Mill Method," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 119, p. 330 (1936).

Test Procedure

5. (*a*) A 500-g. sample of the air-dried coal sized between the 1680-micron (No. 12) and 74-micron (No. 200) sieves (Section 4) shall be obtained by riffling. The charge of 100 balls shall be placed in the mill and the 500-g. sample added. Then the mill shall be closed and rotated for 50 revolutions at a speed of 40 r.p.m. The contents of the mill shall then be poured onto a sieve coarse enough to retain the balls but not the coal. The mill and balls shall be brushed to remove the coal. The sample shall be sieved using either the hand sieving or the mechanical sieving method.

(*b*) For hand sieving, the product shall be placed on a 10-in., 74-micron (No. 200) sieve and shaken until not more than 0.5 g. passes in 1 min. of sieving. Certain coals of a sticky nature are difficult to sieve. With such coals, hand sieving is best done by rocking the sieve back and forth briskly on a table top to break up agglomerates and to prevent clogging of the openings.

(*c*) For mechanical sieving, the product of the ball mill shall be divided into two or more approximately equal parts and each part shall be placed on an 8-in. 74-micron (No. 200) sieve, each provided with a pan. The sieves shall then be shaken for 10 min. in the sieving machine, after which the sieves shall be removed from the pan and the underside of each carefully cleaned with a 1-in. bristle brush in such a manner that any coal removed falls into the pan. The sieves and pans shall then be returned to the sieving machine for an additional 5-min. period of sieving.

(*d*) After sieving, the total undersize shall be weighed and the number of revolutions of the mill required to increase the weight of the undersize to 50 g. shall be calculated by direct proportion. This number of revolutions constitutes a "cycle." Both the oversize and the undersize shall be returned to the mill and it shall be rotated for the additional number of revolutions required to complete the cycle. Again the mill shall be removed from the frame and the contents screened on the 74-micron (No. 200) sieve as before. The undersize product including the loss, which usually ranges from 1 to 3 g. and is assumed to belong to the undersize, shall weigh between 45 and 55 g. These operations comprise the first cycle. For the second cycle the oversize only shall be returned to the mill and ground for the number of revolutions previously found necessary to give approximately 10 per cent of undersize. The material finer than the 74-micron (No. 200) sieve shall be removed as before. The same procedure of alternate milling of the oversize and removal of the undersize shall be continued with the same number of revolutions per cycle until at least 80 per cent or 400 g. of the original sample passes the 74-micron (No. 200) sieve. The end point of the test shall be considered as the number of revolutions of the mill required to grind exactly 80 per cent of the original sample to pass the 74-micron (No. 200) sieve. This exact number of revolutions shall be determined by interpolation.

Number of Tests

6. Duplicate tests shall be made. In the second test the preliminary step taken in the first test to determine the number of revolutions necessary to give 50 g. of undersize shall be omitted. The number of revolutions per cycle shall be the same in both tests.

Reproducibility of Results

7. In duplicate tests made in the same laboratory the number of revolutions required shall agree within 4 per cent.

Expression of Results

8. Results shall be expressed as the average number of revolutions required to grind 80 per cent of the sample to pass the 74-micron (No. 200) sieve or as follows:

$$\text{Ball-mill grindability index, per cent} = \frac{50{,}000}{\text{Average number of revolutions}}$$

Rapid Control Modification

9. For control work on coals having grinding characteristics that have been determined by complete tests, or for large-scale investigations, it shall be considered permissible to shorten the method by taking the endpoint as the number of revolutions required to grind 40 per cent or 200 g. of the original sample to pass the 74-micron (No. 200) sieve. When the shortened procedure is used the results shall be expressed in terms of 80 per cent or 400 g. passing the 74-micron (No. 200) sieve by extrapolation from the number of revolutions of the mill required to grind 40 per cent of the sample to pass the 74-micron (No. 200) sieve. Extrapolation shall be made by the following formula:

$$R = (2 + X)\,N + Y$$

where N = the number of revolutions of the mill required to grind 40 per cent of the sample to pass the 74-micron (No. 200) sieve, and

R = the calculated number of revolutions required to grind 80 per cent of the sample to pass the 74-micron (No. 200) sieve.

The proper values of X and Y to give the correct value of R shall be determined by the operator's knowledge of the grinding characteristics of the coal. For many coals X has the value 0.055 and Y equals 47.[1]

Results of shortened tests shall be clearly designated "Ball-mill grindability index by rapid control modification."

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[1] For information concerning these constants, see discussion by H. L. Brunjes of paper on "Further Investigation of Methods for Estimating the Grindability of Coal," by H. F. Yancey and M. R. Geer, *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 119, p. 371 (1936).

APPENDIX

Approximate conversions of ball-mill grindability indexes into Hardgrove grindability indexes, as determined by the Tentative Method of Test for Grindability of Coal by the Hardgrove-Machine Method (A.S.T.M. Designation: D 409 - 37 T) of the American Society for Testing Materials,[1] may be made by means of a curve plotted from equivalent values, as follows:

BALL-MILL GRINDABILITY INDEX, PER CENT	EQUIVALENT HARDGROVE GRINDABILITY INDEX
20	29
30	43
40	56
50	68
60	80
70	90
80	100
90	110
100	118

According to available data, this conversion of ball-mill grindability indexes into Hardgrove grindability indexes should be accurate to approximately ±3 units for coals having ball-mill grindability indexes ranging from 25 to 50. Sufficient data are not available to determine the accuracy of conversion for coals falling outside this range.

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[1] See p. 815.

TENTATIVE METHOD OF TEST

FOR

GRINDABILITY OF COAL BY THE HARDGROVE-MACHINE METHOD[1]

A.S.T.M. Designation: D 409 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1935; REVISED, 1937.

Scope

1. This method[2] is used to determine the relative grindability or ease of pulverizing of coals in comparison with a coal chosen as 100 grindability. The method is based on Rittinger's Law, which states: "The work done in pulverizing is proportional to the new surface produced." A prepared sample receives a definite amount of grinding energy in a miniature pulverizer and the new surface is determined by sieving.

Apparatus

2. The apparatus for the grindability test shall consist of the following:

(*a*) *Grindability Machine.*—A grindability machine such as is shown in Fig. 1 is required for this test. The eight 1-in. balls roll on a stationary ring and are driven from above by a rotating ring. The action of the rolling balls causes an increase in the surface of the sample being tested. A definite pressure of 64 lb. ± $\frac{1}{2}$ lb. on the balls is obtained by the weights, shaft, top grinding ring and gear. A predetermining counter is used to stop the motor automatically as soon as the vertical shaft of the grindability machine has made exactly 60 revolutions.

(*b*) *Sieves.*—The following sieves will be required: 1190-micron (No. 16), 590-micron (No. 30), and 74-micron (No. 200). The sieves shall conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3]

(*c*) *Mechanical Sieving Device.*—A mechanical sieving device is desirable, although not necessary. If a mechanical sieving device is not avail-

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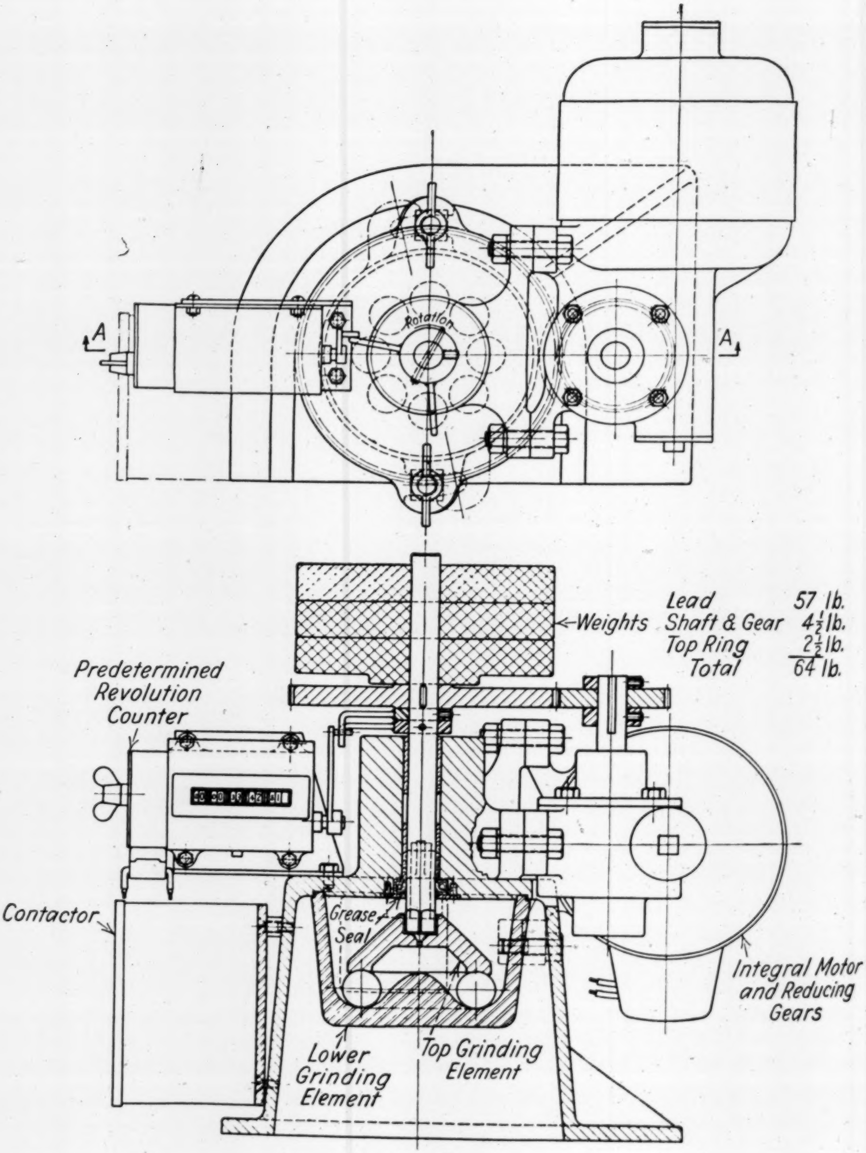
[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke.

[2] For information concerning the experimental work on which this method is based, see paper by R. M. Hardgrove, "Grindability of Coal," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 54, F.S.P., p. 37 (1932).

[3] 1936 Book of A.S.T.M. Standards, Part II, p. 1413.


Rotation
A
A
Lead 57 lb.
Shaft & Gear 4½ lb.
Top Ring 2½ lb.
Total 64 lb.
Weights
Predetermined Revolution Counter
Contactor
Grease Seal
Lower Grinding Element
Top Grinding Element
Integral Motor and Reducing Gears

Section "A-A"

FIG. 1.—Hardgrove Grindability Machine.

able, it will be necessary to give the sample an equivalent amount of hand sieving.

(*d*) *Balance.*—A triple beam balance having a sensitivity of 1 cg., and with which weighings from 1 cg. to 111 g. can be made, is well adapted for this test. However, a torsion or trip balance with suitable pan, and of the specified sensitivity may be used. Greater sensitivity than 1 cg. is not necessary for this test.

(*e*) *Laboratory Crusher.*—The sizing of the sample to be placed in the grindability machine shall be such that it will pass the 1190-micron (No. 16) sieve, and remain on the 590-micron (No. 30) sieve. In order to break up the coarser particles of the sample being prepared for test, a laboratory crusher or a coffee mill will be found desirable. The use of such a device allows the reduction of the coarser particles without the production of excess fine material.

Gross Sample

3. A representative gross sample of coal shall be collected and prepared in accordance with the Standard Method of Sampling Coal (A.S.T.M. Designation: D 21) of the American Society for Testing Materials.[1]

Preparation of Sample

4. An air drier and a riffle sampler, as described in Section 1 (*a*) and (*h*), respectively, of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271) of the American Society for Testing Materials,[2] or equivalent are desirable in preparing the sample. The laboratory sample, after air drying until the loss in weight is not more than 0.1 per cent per hour, shall be placed on the 1190-micron (No. 16) sieve nested with a 590-micron (No. 30) sieve and bottom pan. The sieves shall be shaken by a mechanical sieving machine for approximately 2 min., or given an equivalent amount of hand sieving. The material remaining on the 1190-micron (No. 16) sieve shall be put into a laboratory crusher or a coffee mill in which the set screw has been adjusted so that the coarsest particles are broken up. The sample after passing through the laboratory crusher or coffee mill shall be returned to the 1190-micron (No. 16) sieve of the nest of sieves and again shaken by the mechanical sieving machine or hand sieved. This operation shall be repeated several times until the entire sample has passed through the 1190-micron (No. 16) sieve. The material remaining on the 590-micron (No. 30) sieve shall then be shaken to thoroughly remove any material smaller than this size. All material passing the 590-micron (No. 30) sieve shall be discarded. By using this method of preparing the sample, a minimum of fines is produced.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 382.

[2] 1937 Supplement to Book of A.S.T.M. Standards, p. 153.

Operation of Grindability Machine

5. The predetermined counter shall be set so that the machine will automatically stop after 60 revolutions have been completed. To insure stoppage of the machine within one quarter of one revolution, two trippers are furnished. One tripper is fixed, and the other may be set to correspond with the coasting of the machine after the switch is opened. Therefore, the number of counts on the counter will be 120, or just twice the number of revolutions made by the machine. The grinding elements shall be thoroughly cleaned by brushing before the sample to be tested is added. The eight 1-in. diameter balls shall be placed in the bottom of the lower grinding element and 50 g. of the prepared No. 16 to No. 30 size sample shall be distributed evenly over the balls. Any material falling on the elevated section of the lower grinding element shall be brushed towards the balls. The upper grinding element shall then be placed on the balls and the lower end of the shaft shall be fitted to the square opening in the disk and the grinding elements securely fastened in place by two thumb screws on the sides of the lower grinding element. Care shall be taken to pull the bowl up evenly with the thumb screws. The set screw on the collar which supports the driving gears shall always be kept tight, otherwise the weights will rest on the frame and there will be no pressure on the balls. The predetermining counter shall be set to zero, and the starting switch closed. After the machine has been automatically stopped, the sample shall be transferred to the 74-micron (No. 200) sieve.

Sieving

6. The 74-micron (No. 200) sieve shall be shaken by a mechanical sieving machine for 10 min., or given an equivalent amount of hand sieving. The sieve shall then be removed and the underside carefully cleaned with a 1-in. bristle brush in order to remove any adherent material. The sample shall then be shaken by the mechanical sieving machine for 5 min. more or given an equivalent amount of hand sieving, after which the brushing of the underside of the sieve shall be repeated. After an additional 5 min. of sieving, the material is ready for weighing. A total of 20 min. of mechanical sieving, or an equivalent amount of hand sieving, should result in a comparatively clean sieve.

Weighing the Sample

7. Coals having a high residual moisture content (this is especially true of lignites) lose some of their residual moisture when in a pulverized condition. For this reason rapid and fairly accurate weighing is essential. The material passing through the 74-micron (No. 200) sieve shall be discarded and the material retained on the sieve shall be weighed to within 0.1 g. and the weight recorded.

Calculation of Results

8. The grindability index shall be calculated from the following equation:

$$\text{Hardgrove grindability index}^1 = 13 + 6.93\,W$$

where W = the weight of material passing the 74-micron (No. 200) sieve, determined from the weight of the original sample (50 g.) minus the weight of the material retained on the 74-micron (No. 200) sieve.

Reproducibility of Results

9. The permissible variation between two or more determinations shall not exceed the following:

	PER CENT
Same laboratory	2
Different laboratories	3

APPENDIX

Approximate conversions of Hardgrove grindability indexes into ball-mill grindability indexes, as determined by the Tentative Method of Test for Grindability of Coal by the Ball-Mill Method (A.S.T.M. Designation: D 408 - 37 T) of the American Society for Testing Materials,[2] may be made by means of a curve plotted from equivalent values, as follows:

HARDGROVE GRINDABILITY INDEX	EQUIVALENT BALL-MILL GRINDABILITY INDEX, PER CENT
20	14
30	21
40	28
50	36
60	44
70	52
80	60
90	70
100	80
110	90

According to available data, this conversion of Hardgrove grindability indexes into ball-mill grindability indexes should be accurate to approximately ±3 units for coals having Hardgrove grindability indexes ranging from 35 to 70. Sufficient data are not available to determine the accuracy of conversion for coals falling outside this range.

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[1] The calibration of the machine may be checked by running samples of low volatile run-of-mine bituminous coal from the Jerome Mine, Upper Kittanning Bed, Somerset County, Pa. The average of three tests should give a Hardgrove grindability index of 100 ± 2.

[2] See p. 810.

TENTATIVE METHOD OF DROP SHATTER TEST FOR COAL[1,2]

A.S.T.M. Designation: D 440 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1937.

Scope

1. (*a*) This method of drop shatter test for coal is intended for measuring the relative size stability, and its complement the friability, of lump coal. It affords a means of indicating the ability of coal to withstand breakage when subjected to handling at the mine and during its transit to the consumer. The method of test is considered applicable for testing both a standard size of different coals and for testing different sizes of the same coal, 2 to 3 in. being recommended as the standard size for testing different coals. The method includes separate procedures for, (*a*) testing the 2 to 3-in. standard single size, and (*b*) a supplementary method for other single sizes and for mixed sizes of the same coal (see Section 9 and Explanatory Note 1).

(*b*) The method appears best suited for measuring the relative resistance to breakage of the larger sizes of lump coal when handled in thin layers such as from loader to mine car, from loading boom to railroad car, from shovel to chute, etc. While it may not be so well adapted for measuring the liability to breakage of coal when handled in mass, as in unloading open-bottom cars, emptying bins, etc., it is believed that the method of test and particularly the supplementary method will serve to indicate the relative size stability of composite sizes of coal where, in commercial handling, the smaller sized particles and lumps have a cushioning effect which tends to lessen the breakage of the larger lumps of coal.

Method of Test for 2 to 3-in. Coal as Standard Size

Shatter Test Machine

2. The shatter test machine, which is the same as that described and illustrated in the Standard Method of Shatter Test for Coke (A.S.T.M.

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[1] **Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke.**

[2] **For information concerning the development and utilization of this drop shatter test method for coal the following references may be consulted:**

C. M. Smith, "An Investigation of the Friability of Different Coals," University of Illinois, Engineering Experiment Station, *Bulletin No. 196* (1929); "The Friability of Illinois Coals," University of Illinois, Engineering Experiment Station, *Bulletin No. 218* (1930).

H. F. Yancey and R. E. Zane, "Comparison of Methods for Determining the Friability of Coal," U. S. Bureau of Mines, *Report of Investigations 3215* (1933).

R. E. Gilmore, J. H. H. Nicolls and G. P. Connell, "Coal Friability Tests," Canadian Department of Mines, Mines Branch, *No. 762* (1935).

Designation: D 141) of the American Society for Testing Materials,[1] shall consist of a box 18 in. in width, 28 in. in length, and approximately 15 in. in depth, supported above a rigidly mounted cast-iron or steel plate, not less than ½ in. in thickness, 38 in. in width, and 48 in. in length. The inside of the bottom of the box shall be 6 ft. above the plate. The bottom of the box shall consist of two doors hinged lengthwise and latched so that they will swing open freely and not impede the fall of the coal. Boards about 8 in. in height should be placed around the plate so that no coal is lost. To prevent the breakage of coal, which may occur while placing the sample in the box, the box shall be constructed so that it can be lowered to a convenient level; this is best done by means of a pulley and counterweight. A convenient form of shatter test machine is shown in Fig. 1.

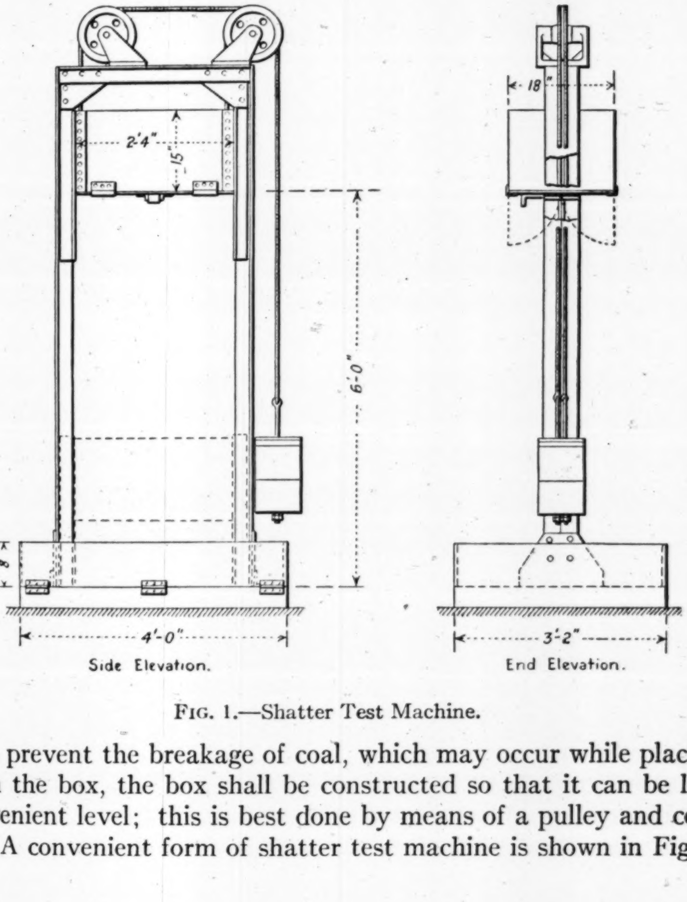

FIG. 1.—Shatter Test Machine.

Screens

3. Round-hole screens having 3, 2, 1½, 1, ¾, and ½-in. diameter openings shall be used. These screens shall conform to the Standard Specifications for Round-Hole Screens for Testing Purposes (A.S.T.M. Designation: E 17) of the American Society for Testing Materials.[2] Frames for the

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 449.

[2] *Ibid.* p. 1411.

screens may be of either hardwood or metal, and may be square, rectangular, or circular. A nest comprising all the screens in the series, with 2-ft. square plates, that is, of 4-sq. ft. area, is to be recommended, although plates with areas of 6 to 9 sq. ft., either rectangular or square, are suitable.

Collection of Gross Sample

4. The gross sample shall be obtained in accordance with Sections 4 and 5 of the Tentative Method of Test for Screen Analysis of Coal (A.S.T.M. Designation: D 410 - 35 T) of the American Society for Testing Materials.[1] In order that the entire quantity of the coal sampled will be represented proportionately in the gross sample, increments shall be regularly and systematically collected. When testing lump coal as mined, the sample shall be taken at the mine before it is subjected to screening and to loading into cars at the tipple. When testing coals subsequent to mining, the sample may be taken at any stage in the transportation from the mine to the place at which it is to be used. For the correct interpretation of the shatter test results, especially if the coal tested has been subjected to weathering, the elapsed time since mining as well as a record of the handling and storage of the coal shall be noted.

Preliminary Screening and Preparation of 2 to 3-in. Size

5. Using the screens designated in Section 3, a preliminary screening of a representative portion, or all, of the gross sample shall be made and the screen sizes retained separately. Successive representative portions of the gross sample shall be screened to obtain at least 125 lb. of the 2 to 3-in. size desired for test. In cases where difficulty is experienced in screening out this quantity, the amount obtained by the preliminary screening may be augmented from larger lumps, either by breaking them with a hammer or by dropping them in the shatter test apparatus. The obtaining in this way from larger lumps of an adequate quantity of a particular size selected for test is especially applicable to freshly mined lump coal.

Rescreening of Sample for Test

6. The total quantity of the 2 to 3-in. size, obtained as described in Section 5, shall be thoroughly mixed and then rescreened to pass the 3-in. screen and be retained on the 2-in. screen. Only a thin layer of coal shall be placed on the screen so as to allow the pieces to be in direct contact with the screen openings. Individual pieces of coal not passing readily through either of the screens shall be tried by hand to see if they will pass through the openings in any position without forcing.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 863 (1935); also 1937 Book of A.S.T.M. Tentative Standards, p. 628.

Procedure 50-lb. Sample Dropped Twice

7. (*a*) A 50-lb. portion of the coal sample prepared in accordance with Sections 5 and 6 shall be given two drops. The coal shall be placed in the box of the shatter test machine, the coal leveled, and then dropped a distance of 6 ft. onto the plate. All the coal on the plate shall be carefully returned to the box and again dropped. After the second drop the material shall be successively run through the screens specified in Section 3. In screening, care shall be taken to prevent further breakage of the coal. The screening shall be carried out in such small increments as to permit satisfactory contact between the individual pieces of coal and the screen. On the three larger screens, 2, 1½, and 1 in., individual pieces of coal not readily passing through the screens shall be tried by hand to see if they will pass through the openings in any position without forcing. When using the screens with ¾-in. openings and smaller, the coal shall come into intimate contact with the screen either by shaking or rolling by hand without up-ending the individual lumps.

TABLE I.

SCREEN ANALYSIS OF COAL USING ROUND-HOLE SCREENS

RETAINED ON	PASSING	WEIGHT, RECORDED, LB.	WEIGHT, PER CENT (*1*)	AVERAGE OF SCREEN OPENINGS INCHES (*2*)	AVERAGE OF SCREEN OPENINGS FACTOR (*3*)	PRODUCT OF (*1*) × (*3*)
Sample:						
2 in.	3 in.	50	100.0	2.500	1	100 = S
Dropped Coal:						
2 in.	3 in.	33	66.0	2.500	1	66.000
1½ in.	2 in.	6¼	12.5	1.750	0.7	8.750
1 in.	1½ in.	4	8.0	1.250	0.5	4.000
¾ in.	1 in.	1¾	3.5	0.875	0.35	1.225
½ in.	¾ in.	1½	3.0	0.625	0.25	0.750
	½ in.	3½	7.0	0.250	0.1	0.700
Total (Sum of products (*1*) × (*3*) for dropped coal)						81.425 = s

$$\text{Size stability, per cent} = \frac{100 \times s}{S} = \frac{100 \times s}{100} = s = 81.4.$$

To be reported as: Size stability, 81.5 per cent.

(Friability, per cent, would be: 100 – 81.5 = 18.5.)

(*b*) The coal remaining on each screen, and that which passes through the bottom screen shall be weighed either separately or in cumulative portions, on a scale sensitive to ¼ lb. or less. By the cumulative portion method the largest 2 to 3-in. sized lumps shall be weighed into a tared container, which container shall then be reweighed after addition of the successive individual smaller screened sizes of dropped coal. If the final net weight so obtained, or the sum of the separate weights, shows a loss of over 1 per cent, the test shall be rejected and another made, the loss in each case to be included in the percentage passing the ½-in. screen. At least two tests shall be made to obtain size stability results agreeing within 2 per cent. When three or more tests are considered advisable and are made, all the results within a maximum to minimum limit of 3 per cent may be averaged.

Reporting of Results

8. The percentage weight screen analysis shall be reported to the nearest 0.1 per cent, and the percentage size stability to the nearest 0.5 per cent (Note).

NOTE.—A numerical example of tabulating the results and of calculating size stability, per cent, is shown in Table I, where the average of the screen size openings limiting the different sizes is expressed to the nearest 0.005 in. The data shown are for a typical, medium friable coal.

Supplementary Method of Test for Other "Single" Sizes and "Mixed" Sizes

Scope

9. This supplementary method is applicable to both single sizes other than the 2 to 3-in. standard size and to mixed sizes of coal. By single sizes are meant those with fixed limits of retaining and passing screen openings and by mixed sizes are meant either slack coal or a mixture of two or more single sizes (see Explanatory Note 1).

Shatter Test Machine

10. The shatter test machine to be employed shall be that described in Section 2.

Screens

11. The list of screens specified in Section 3 shall be supplemented by screens having $\frac{3}{8}$, $\frac{1}{4}$, and $\frac{1}{8}$-in. diameter openings for testing coal smaller than the 2 to 3-in. standard size. For larger lumps of coal other screens or rings with 4, 6, and 8-in. openings shall also be used.

Collection of Gross Sample

12. The gross sample of coal shall be obtained in accordance with Section 4.

Preparation of Sample

13. The sample of coal for test shall be prepared in accordance with Sections 5 and 6. For slack coals and mixed sizes the sample shall be carefully prepared either by process of quartering or by reassembling the different sizes in the proportion obtained in the preliminary screening of the lot of coal to be tested. For $\frac{3}{4}$-in. and smaller size slack coals, quartering is satisfactory, while for larger size slack coals and for blends of two or more single sizes the latter reassembling method is recommended. Before dropping, the sample shall be rescreened on the same series of screens selected for screening the dropped coal.

Procedure

14. The procedure for testing 50-lb. samples of single sizes other than the 2 to 3-in. standard size and for mixed sizes shall be essentially the same as outlined in Section 7. When testing sizes larger than 2 to 3 in., the screens to be used for the dropped coal shall be selected from the list shown in Section 15 down to and including the screen with ½-in. openings. The screens with ⅜, ¼, and ⅛-in. openings are optional and supplementary for testing samples smaller in size than ¾ in. When testing the larger size lumps it is recommended that after dropping two individual 50-lb. samples twice, as required, the screen analysis may be made on 100 lb. of dropped coal instead of screening each 50 lb. separately. In each case the result shall be checked by dropping and screening a second two lots of 50 lb. each, provided sufficient sample is available. This variation in procedure is specially recommended for lumps larger than 8 in., for which size it is advisable that as many as ten lots of approximately 50 lb. each be tested and the average result reported.

Reporting of Results

15. The percentage weight screen analysis shall be reported to the nearest 0.1 per cent and the size stability per cent to the nearest 0.5 per cent. The method of recording and calculating the results may follow the numerical example shown in Section 8, altered to suit the particular size tested, or may be in accordance with the tabulated form shown below:

SCREEN ANALYSIS OF COAL BEFORE AND AFTER TEST (TWO DROPS) ROUND-HOLE SCREENS		WEIGHT, PER CENT		AVERAGE OF SCREEN SIZE OPENINGS, INCHES	PRODUCT OF WEIGHT PERCENTAGE AND OF SCREEN OPENINGS	
RETAINED ON	PASSING	BEFORE TEST	AFTER TEST		BEFORE TEST	AFTER TEST
8 in.						
6 in.	8 in.			7.000		
4 in.	6 in.			5.000		
3 in.	4 in.			3.500		
2 in.	3 in.			2.500		
1½ in.	2 in.			1.750		
1 in.	1½ in.			1.250		
¾ in.	1 in.			0.875		
½ in.	¾ in.			0.625		
⅜ in.	½ in.			0.435		
¼ in.	⅜ in.			0.315		
⅛ in.	¼ in.			0.185		
	⅛ in.			0.060		
Total passing	⅜ in.			0.185		
Total passing	¼ in.			0.125		
Average size of coal before and after test (2 drops)............					Total (*S*)	Total (*s*)

$$\text{Size stability, per cent} = \frac{(s) \times 100}{(S)} = \text{..........................}$$

(Friability, per cent, will be 100 – size stability, per cent.)

EXPLANATORY NOTES

NOTE 1.—When reporting the results of the drop shatter test it shall be clearly stated to which single size or mixed sizes of coal the size stability, per cent, applies, whether to the 2 to 3-in. standard size or to other larger or smaller sizes. The following form may serve as a guide.

Size Designation of Sample		Size Stability, per cent	Slack Index
Single sizes larger than standard	8 in. and larger		
	6 to 8 in		
	4 to 6 in		
	3 to 4 in		
Standard size	2 to 3 in		
Single sizes smaller than standard	$1\frac{1}{2}$ to 2 in		
	1 to $1\frac{1}{2}$ in		
	$\frac{3}{4}$ to 1 in		
Mixed sizes	$\frac{3}{4}$ to 4 in		
	$\frac{3}{4}$-in. slack		

NOTE 2.—For drop shatter tests on the single sizes in addition to the size stability, per cent, the percentage passing the screen with the $\frac{3}{4}$-in. openings may be reported to the nearest whole per cent as the "slack index" to indicate the comparative slack producing characteristics of the particular size of the coal tested, for example: Size stability, per cent, of 2 to 3-in. lumps ....; with slack index of ....

TENTATIVE METHOD OF TUMBLER TEST FOR COAL[1,2]

A.S.T.M. Designation: D 441 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. This method of tumbler test for coal is intended for determining the relative friability of a particular size of lump coal. It affords a means of measuring the liability of coal to break into smaller pieces when subjected to repeated handling at the mine, or subsequently by the distributor or by the consumer. It also may serve to indicate the relative extent to which lump coals will suffer size degradation in certain mechanical feed devices. Furthermore, the test may be employed for differentiating between certain ranks and grades of coal, and hence be of service for coal classification purposes.

Porcelain Jar Tumbler

2. The tumbler shall consist of a cylindrical porcelain jar of uniform dimensions, $7\frac{1}{4}$ in. in depth and $7\frac{1}{4}$ in. in diameter, inside measurements, such as is employed for pulverizing coal samples for analysis. The jar shall be fitted inside with an iron frame with lifting shelves constructed as shown in Fig. 1. The two rings, *a*, shall be $7\frac{1}{8}$ in. in outside diameter and shall be made of $\frac{3}{4}$ by $\frac{1}{8}$-in. iron. The three ledges or shelves, *b*, $6\frac{1}{2}$ by $\frac{3}{4}$ by $\frac{1}{8}$ in., shall be attached radially to the rings by means of small brackets, *c*, the ends of the shelves being flush with the outer edges of the rings. The shelves shall be attached so that there will be $\frac{5}{8}$-in. clearance between their outer edge and the outside of the ring. Rivets, not bolts, shall be used in constructing the frame. As the jars available commercially are not of absolutely uniform size, the measurements of the frame may be

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke.

[2] For information concerning the development and utilization of this tumbler test method for coal, the following references may be consulted:

J. H. H. Nicolls, "Friability Tests on Various Fuels Sold in Canada," Paper II, Canadian Department of Mines, Mines Branch, *No. 644* (1926).

I. Lavine, A. W. Gauger and C. A. Mann, "Studies in the Development of Dakota Lignite," *Industrial and Engineering Chemistry*, Vol. 22, No. 12, p. 1360 (1930).

C. E. Lawall and C. T. Holland, "Some Physical Characteristics of West Virginia Coals," Am. Inst. Mining and Metallurgical Engrs., Coal Division, Vol. 101, p. 100 (1930).

H. F. Yancey, K. A. Johnson, and W. A. Selvig, "Friability, Slacking Characteristics, Low Temperature Carbonization Assay and Agglutinating Value of Washington and Other Coals," U. S. Bureau of Mines, *Technical Paper No. 512* (1932).

H. F. Yancey and R. E. Zane, "Comparison of Methods for Determining the Friability of Coal," U. S. Bureau of Mines, *Report of Investigations 3215* (1933).

R. E. Gilmore, J. H. H. Nicolls and G. P. Connell, "Coal Friability Tests," Canadian Department of Mines, Mines Branch *No. 762* (1935).

slightly varied to suit individual cases. The frame shall be fixed inside the jar, by means of wedges between the rings and the inside wall of the jar, so that its axis shall coincide as nearly as possible with the axis of the jar, and so that the frame will rotate with the jar. The jar shall be closed by a set-in porcelain lid, resting upon a heavy rubber gasket, and sealed tightly according to the customary procedure with such jars, that is, by means of a bolt working against the lid. The bolt shall be set in a crossbar, the ends of which shall be held by a metal strip which fits around the body of the jar. For tumbling, the jar shall be laid in a horizontal position, in a suitable support or rack and rotated about its cylindrical axis at the rate of 40 r.p.m. The assembled apparatus is illustrated in Fig. 2.

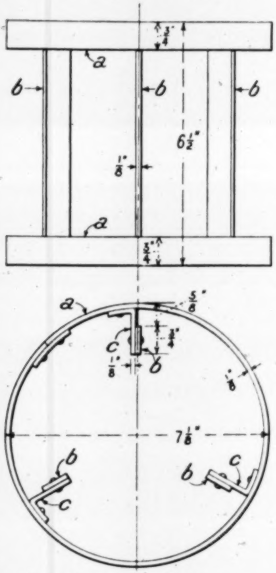

FIG. 1.—Steel Frame Used in Jar Mill.

Optional Iron Jar Tumbler

3. An iron jar may be substituted for the porcelain jar specified in Section 2, providing it has approximately the same internal dimensions, namely, 7¼ in. in diameter by 7¼ in. in depth. A jar constructed of cast iron is recommended, the interior of which shall be machined to the required internal dimensions. For making it sufficiently light for lifting, the jar may have a wall thickness of not less than ¼ in., except at each end. It is recommended that the lid, the rubber gasket, and the metal strip which passes from the bottom up the outside of the jar to serve in holding in place the crossbar above the lid, be similar in design with those for the porcelain jar. The wall of the iron jar for a distance of approximately 1 in. from

the top shall have a thickness of at least $\frac{1}{2}$ in. to correspond with that of the porcelain jar; and in order that the metal strip may fit evenly, the lower inch of the wall should also have a thickness of not less than $\frac{1}{2}$ in.

Screens (or Sieves)

4. (*a*) For sizing the sample for test, square-mesh screens having 1.5 and 1.05-in. actual openings between the wires shall be used. These screens may be fitted into frames, 12 by 30 in. or larger.

(*b*) For screening the coal after tumbling, square-mesh screens or sieves having 1.05, 0.742, 0.525, 0.371, 0.0469, and 0.0117-in. actual openings between the wires shall be used, the last two sieves being No. 16 and No. 50 of the U.S. standard sieve series conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[1] For this purpose, round, metal-framed screens 8 in. in diameter are suitable.

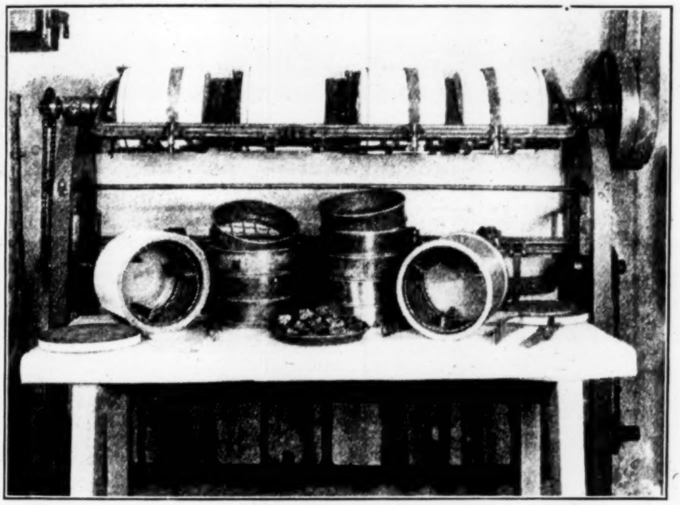

FIG. 2.—Tumbler Test Apparatus.

On the bench are shown typical jar mills in which the three-vane iron frames are fitted, together with a set of 8-in. diameter screens and a 1000-g. charge of 1.05 to 1.5 in. lumps of coal. The jar to the left is one of the regular porcelain jars, and that to the right an optional iron jar.

Collection of Gross Sample

5. The gross sample of coal shall be obtained in accordance with Sections 1 to 4, inclusive, and the directions preceding Section 1, of the Standard Method of Sampling Coal (A.S.T.M. Designation: D 21) of the American Society for Testing Materials.[2]

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

[2] *Ibid.*, p. 382.

Preparation of Sample

6. (*a*) Using square-hole screens with 1.05 and 1.5-in. openings, a preliminary screening of a representative portion, approximately 100 lb., of the gross sample shall be made.

(*b*) Should the preliminary screening (Paragraph (*a*)) indicate that the lumps are mostly smaller than 1.5 in., successive representative portions of the gross sample shall be screened until at least 25 lb. of the 1.05 to 1.5-in. lumps are obtained.

(*c*) Should it be found that most of the lumps obtained by the preliminary screening (Paragraph (*a*)) are larger than 1.5 in., screening of representative portions of the gross sample shall be conducted until at least 50 lb. of the screened material remaining on the 1.05-in. screen are available. The lumps so obtained that are larger than 1.5 in. shall then be broken with a hammer until they pass the 1.5-in. screen, avoiding as much as possible the production of lumps smaller than 1.05 in. Should the combined weights of the 1.05 to 1.5-in. size, obtained by screening before and after hammer breakage of the larger lumps, be less than the required 25 lb., the amount shall be augmented by further screening and breakage of additional representative portions, or the remainder of the gross sample, care being taken to discard all lumps in which noticeable cracks have developed by hammer breakage. Care shall also be taken to provide lumps covering the whole range of the 1.05 to 1.5-in. size in both the 25-lb. composite sample, and in each 1000-g. sample taken for test as in Section 8. This may be accomplished by the use of a 1.25-in. screen to see that approximately half of the weight of the lumps comprising these samples will be between 1.05 and 1.25-in. and half between 1.25 and 1.50-in.

Rescreening of Sample for Test

7. The total quantity of the 1.05 to 1.5-in. size, obtained as described in Section 6, shall be thoroughly mixed and then rescreened to pass the 1.5-in. screen and be retained on the 1.05-in. screen. Only a thin layer of coal shall be placed on the screen so as to allow the pieces to be in direct contact with the screen openings. Individual pieces of coal not passing readily through the screen shall be tried by hand to see if they will pass through the openings in any position without forcing

Procedure

8. Approximately 1000 g. of the coal sample prepared in accordance with Sections 6 and 7 shall be weighed and placed in the jar, and the jar rotated in the tumbler test machine at 40 ± 1 r.p.m. for 1 hr. In order to standardize the time of tumbling, a revolution counter should be used, either periodically or as permanent accessory equipment to the machine, to ensure that the total number of revolutions during a test is approximately 2400. After tumbling, the coal shall be thoroughly graded as to

size upon the screens and sieves designated in Section 4. The screening shall be carried out in such small increments as to permit satisfactory contact between the individual pieces of coal and the screen. On the two larger screens, 1.05 and 0.742 in., individual pieces of coal not readily passing through the screens shall be tried by hand to see if they will pass through the openings in any position without forcing. Screening may be carried out either by hand or mechanically, though the former method is preferable. At least four single-jar tests shall be made, and, provided sufficient sample is available, it is recommended that two or more four-jar tests be made. When only four single-jar tests are made, the contents of each jar shall be screened separately in order to be sure that there is satisfactory agreement between the results obtained. When two or more four-jar tests are made the contents of the four jars from each set may be mixed and screened together. The weighings shall be made on a balance sensitive to 1 g.

Reporting of Results as Friability, Per Cent

9. The results of test shall be reported to the nearest 0.5 per cent as friability, per cent, which is the percentage reduction in average size of the coal during the tumbling test (Note).

NOTE.—A numerical example of the method of calculating friability is given in tabular form below, where the average of the openings of the retaining and passing screens is expressed in inches to the nearest 0.001 in. The data shown are for a typical, medium friable coal. It is from the average of the screen openings that the approximate relative size factors, shown as column (3), are derived. In the column to the extreme right S represents the average size of the coal lumps before tumbling and s the average size of the tumbled coal, the value for S being arbitrarily chosen as 100 times its corresponding size factor.

SCREEN ANALYSIS OF COAL USING SQUARE-HOLE SCREENS

RETAINED ON	PASSING	WEIGHT, PER CENT (1)	AVERAGE OF SCREEN OPENINGS INCHES (2)	AVERAGE OF SCREEN OPENINGS FACTOR (3)	PRODUCT OF (1) × (3)	
Sample:						
1.05 in.	1.5 in.	100.0	1.275	1	100.00	= S
Tumbled Coal:						
1.05 in.	1.5 in.	46.2	1.275	1	46.20	
0.742 in.	1.05 in.	26.9	0.896	0.7	18.83	
0.525 in.	0.742 in.	4.0	0.634	0.5	2.00	
0.371 in.	0.525 in.	1.6	0.448	0.35	0.56	
0.0469 in.	0.371 in.	5.5	0.209	0.15	0.825	
0.0117 in.	0.0469 in. (No. 16)	0.5	0.029	0.025	0.010	
	0.0117 in. (No. 50)[a]	15.3[b]	0.006	0.005	0.075	
Total (Sum of products (1) × (3) for tumbled coal)					68.50	= s

$$\text{Friability, per cent} = \frac{100(S - s)}{S} = \frac{100(100 - 68.50)}{100} = 31.5.$$

To be reported as: Friability, 31.5 per cent.

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[a] Including loss, not to exceed 0.5 per cent.

[b] The percentage of "fines and dust" passing the 0.0117-in. (No. 50) screen represents the proportion of the breakage due to attrition or abrasion rather than to shattering, and may be reported as "dust index" to the nearest whole per cent to indicate the relative dust-producing properties of coals when subjected to severe handling. Hence both the friability per cent and the "dust index" may be reported as follows: **Friability, per cent = 31.5; with dust index of 15.**

TENTATIVE METHOD OF TEST

FOR

TAR ACIDS IN CREOSOTE AND CREOSOTE - COAL TAR SOLUTIONS[1]

A.S.T.M. Designation: D 453 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. This method is intended for determining the amount of tar acids in the fractions distilled from creosote and creosote - coal tar solutions.

Apparatus

2. (*a*) *Type I, Separatory Funnel.*—The Type **I** glass separatory funnel shall conform to the requirements shown in Fig. 1 (*a*). It shall have a total capacity of 175 ml. with the lower 20 ml. graduated accurately in 0.1 ml.

(*b*) *Type II, Separatory Funnel.*—The Type II glass separatory funnel shall conform to the requirements shown in Fig. 1 (*b*). The capacity of the lower bulb from the stopcock to the first graduation mark shall be 65 ml., and above this mark the stem shall be graduated accurately for 100 ml. in 0.2 ml.

Procedure

3. (*a*) In making the determination, 100 g. of the sample shall be distilled in accordance with the Standard Method of Test for Distillation of Creosote (A.S.T.M. Designation: D 246) of the American Society for Testing Materials.[2] The fraction (Note) to be tested shall be transferred to a regular 250-ml. glass-stoppered separatory funnel, and 50 ml. of c.p. benzol and 50 ml. of an 18.3 per cent solution of $NaOH$ (sp. gr. 1.20 at 20/40 C.) shall be added. The mixture shall be shaken vigorously for 3 min. and allowed to settle. The well-settled lower portion of the liquid shall then be drawn off into a 250-ml. beaker. An additional 30 ml. of the $NaOH$ solution (18.3 per cent) shall be added to the separatory funnel and

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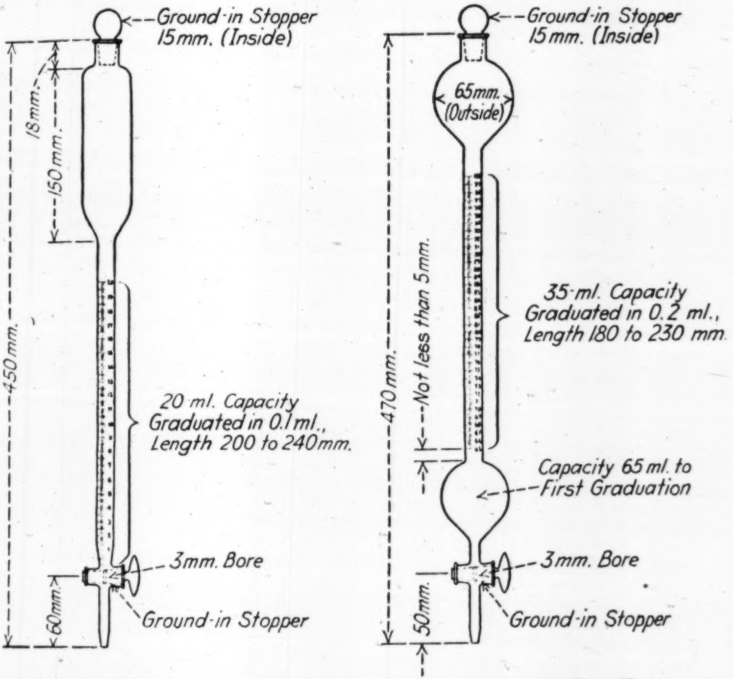
[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 542.

the contents gently shaken for 2 min. After settling, the lower portion of the liquid shall be drawn off and added to the beaker containing the first portion. Sufficient diluted H_2SO_4 (1:3) shall be added to the mixture in the beaker to turn blue litmus paper definitely red. The solution shall be cooled during the addition of the acid.

(*b*) If the tar acids liberated are estimated to be under 10 ml., Type I separatory funnel (Section 2 (*a*)) shall be used and 10 ml. of "Hi-flash" naphtha or c.p. benzol at 25 C. shall be measured into it. The liberated tar acids and sulfate solution shall then be poured through this layer of naphtha or benzol several times, drawing the material off at the bottom of the funnel into the original beaker and pouring it back into the top of the funnel. This washes out the beaker and allows all the tar acids to be absorbed. The funnel shall then be allowed to stand at 25 C., until the layers separate clearly, when the sulfate solution shall be drawn off and the increase in the volume of the naphtha or benzol taken as the dry tar acids present.


Ground-in Stopper 15 mm. (Inside)
18 mm.
150 mm.
450 mm.
20 ml. Capacity Graduated in 0.1 ml., Length 200 to 240 mm.
3 mm. Bore
60 mm.
Ground-in Stopper
Ground-in Stopper 15 mm. (Inside)
65 mm. (Outside)
470 mm.
Not less than 5 mm.
35 ml. Capacity Graduated in 0.2 ml., Length 180 to 230 mm.
Capacity 65 ml. to First Graduation
3 mm. Bore
50 mm.
Ground-in Stopper

(*a*) Type I. (*b*) Type II.

FIG. 1.—Glass Separatory Funnels.

(*c*) If the tar acids liberated are estimated to be more than 10 ml., the procedure described in Paragraph (*b*) shall be followed except that

65 ml. of "Hi-flash" naphtha or c.p. benzol at 25 C. shall be measured into the Type II separatory funnel (Section 2 (*b*)).

NOTE.—If the total content of tar acids in a sample is desired, the entire distillate below 355 C. should be tested. In this case the results shall be reported as percentage by volume, that is, as the number of milliliters of tar acids per 100 ml. of the original dry sample, both volumes measured at the same temperature.

Calculation

4. The amount of tar acids shall be reported as a percentage of the fraction tested, calculated as follows:

$$\text{Percentage of tar acids} = \frac{\text{Volume of tar acids in milliliters}}{\text{Weight of fraction in grams}} \times 100$$

In the above formula, the assumption is made that 1 ml. of tar acids weighs 1 g.

TENTATIVE METHOD OF TEST
FOR
ACID NUMBER OF ROSIN[1]

A.S.T.M. Designation: D 465 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. This method of test is intended for determining the acid number of rosin.

Special Solutions Required

2. (*a*) *Sodium Hydroxide Solution (0.5 N).*—Accurately standardize a 0.5 *N* NaOH solution.

(*b*) *Ethyl Alcohol.*—Ninety-five per cent, neutral, specially denatured alcohol (formula No. 30).

(*c*) *Phenolphthalein Solution.*—Dissolve 1 g. of phenolphthalein in 100 ml. of 95 per cent specially denatured alcohol, and neutralize the slightly acid alcohol solution with a caustic soda solution.

Procedure

3. In a 250-ml. Erlenmeyer flask[2] of heat-resistant glass,[3] dissolve a 1.95 to 2.05-g. ±0.001 g. sample[4] of the rosin in 50 ml. of neutral 95 per cent specially denatured alcohol (formula No. 30). Heat to dissolve the rosin and titrate the hot solution with standard 0.5 *N* sodium hydroxide solution using 1 ml. of phenolphthalein solution as indicator. The standard 0.5 *N* sodium hydroxide solution should be added as rapidly as possible, preferably with a wide-open burette until the end point is approached, and then by rapid dropping until the end point is reached.[5]

Calculation

4. The acid number expressed in milligrams of KOH per gram, shall be calculated from the following formula:

$$\text{Acid Number} = \frac{\text{milliliters of NaOH} \times N \times 56.1}{\text{Weight of sample in grams}}$$

where N = the normality of the sodium hydroxide solution used.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-17 on Naval Stores.

[2] The flasks should be either new or cleaned by rinsing with a hot solution of hydrofluoric acid (2 to 3 per cent). This removes from the flasks the adhering partially disintegrated silicates which would interfere with the determination. This treatment of flasks is not as important for determination of acid number, as for determination of saponification number; however, it should be a good precautionary measure.

[3] Pyrex is very satisfactory for this purpose.

[4] It is desirable to stay within rather narrow limits as to size of sample, in order not to introduce too large variations in the relative concentrations of rosin, alcohol, and alkali solutions.

[5] Rapid titration is desirable to prevent any saponification of neutral constituents which slow titration might entail.

TENTATIVE METHOD OF TEST
FOR
SAPONIFICATION NUMBER OF ROSIN[1]

A.S.T.M. Designation: D 464 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. This method is intended for determining the saponification number of rosin.

Special Solutions Required

2. (*a*) *Hydrochloric Acid Solution* (*0.5 N*).—Accurately standardize a 0.5 *N* HCl solution.

(*b*) *Alcoholic Potassium Hydroxide Solution.*—Dissolve 40 g. of c. p. KOH in 1 liter of 95 per cent redistilled specially denatured alcohol (formula No. 30). The alcohol shall be redistilled from potassium hydroxide over which it has been standing for some time, or with which it has been boiled for some time, under a reflux condenser. The solution must be clear. If it contains insoluble matter, it shall be either decanted after settling all night or filtered.

(*c*) *Phenolphthalein Solution.*—Dissolve 1 g. of phenolphthalein in 100 ml. of 95 per cent specially denatured alcohol (formula No. 30), and neutralize the slightly acid alcohol solution with sodium hydroxide solution.

Procedure

3. Weigh a 2.45 to 2.55-g. ±0.001 g. sample[2] of the rosin into a 250-ml. Erlenmeyer flask[3] of heat-resistant glass.[4] Dissolve the sample in 25 ml. of formula No. 30 alcohol and into it pipette[5] 25 ml. of the alcoholic potassium hydroxide solution, allowing the pipette to drain for a definite time. Connect the flask preferably to a water condenser or to an air condenser made from glass tubing of 5 mm. in inside diameter, having a minimum length of 32 in. Place on a *hot* hot-plate and boil the solution gently for

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-17 on Naval Stores.

[2] It is desirable to stay within rather narrow limits as to size of sample, in order not to introduce too large variations in the relative concentrations of rosin, alcohol, and alkali solutions.

[3] The flasks should be either new or cleaned by rinsing with a hot solution of hydrofluoric acid (2 to 3 per cent). This removes from the flasks the adhering partially disintegrated silicates which would interfere with the determination.

[4] Pyrex glass is very satisfactory for this purpose.

[5] The use of a Lowy automatic pipette is recommended.

exactly 1 hr., taking the starting time at the point of incipient boiling. Cool, and without washing down the condenser, titrate immediately with 0.5 N hydrochloric acid solution using 1 ml. of phenolphthalein indicator.

Conduct two or three blank determinations on 25-ml. portions of No. 30 alcohol with 25-ml. portions of the alcoholic potassium hydroxide solution, using the same pipette and draining for the same length of time as above.

Calculation

4. The saponification number shall be calculated from the following formula:

$$\text{Saponification Number} = \frac{\left(\begin{matrix}\text{ml. of HCl for}\\\text{titration of blank}\end{matrix} - \begin{matrix}\text{ml. of HCl for}\\\text{titration of sample}\end{matrix}\right) \times N \times 56.1}{\text{Weight of sample in grams}}$$

where N is the normality of the standard HCl solution used in the titration.

TENTATIVE SPECIFICATIONS
FOR
BASIC SULFATE WHITE LEAD[1]

A.S.T.M. Designation: D 82 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover the pigment commonly known as "basic sulfate" white lead. The pigment may be purchased in the dry form or ground in oil to form a paste.

Composition

2. (*a*) *Dry Pigment.*—The dry pigment shall conform to the requirements specified in Section 3 (*b*).

(*b*) *Paste.*—The paste shall be made by thoroughly grinding the specified pigment with pure raw or refined linseed oil.

Properties and Tests

3. (*a*) The color and color strength, when specified, shall be equal to that of a sample mutually agreed upon by the buyer and the seller.

(*b*) *Dry Pigment.*—The dry pigment shall conform to the following requirements:

	MAXIMUM	MINIMUM
Coarse particles (total residue retained on a No. 325 sieve[2]), per cent	1.0	....
Lead oxide, per cent	28.0	15.0
Zinc oxide, per cent	5.0	....
Total impurities, including moisture, per cent	1.0	....
Lead sulfate	remainder	

(*c*) *Paste.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile petroleum spirits or any mixture of these substances.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

These tentative specifications are in effect a tentative revision of, and will supersede when adopted as standard, the present Standard Specifications for Basic Sulfate White Lead (A.S.T.M. Designation: D 82 - 24), 1936 Book of A.S.T.M. Standards, Part II, p. 604.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials, see 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

The paste shall conform to the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	....	84.0
Linseed oil, per cent	16.0	....
Moisture and other volatile matter, per cent	0.7	....
Coarse particles and skins (total residue retained on a No. 325 sieve[1]), per cent of the dry pigment	1.5	....

Number of Tests

4. One sample shall be taken at random from each lot of 1000 packages or fraction thereof. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

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[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials, see 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

TENTATIVE SPECIFICATIONS

FOR

TITANIUM-MAGNESIUM PIGMENT[1]

A.S.T.M. Designation: D 442 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1937.

Scope

1. These specifications cover titanium-magnesium pigment in the dry form, also ground in oil to form a paste.

Composition

2. (*a*) *Dry Pigment.*—The dry pigment shall consist of titanium dioxide and magnesium silicates, with or without added aluminum silicates.

(*b*) *Paste.*—The paste shall be made by thoroughly grinding the specified pigment in linseed oil.

Properties and Tests

3. (*a*) The color, brightness, oil absorption and tinting strength of the pigment, when specified, shall be at least equal to that of a sample mutually agreed upon by the buyer and the seller.

(*b*) *Dry Pigment.*—The dry pigment shall conform to the following requirements:

	Maximum	Minimum
Coarse particles (total residue retained on a No. 325 sieve[2]), per cent	2.0	....
Titanium dioxide, per cent	....	28.0
Moisture, per cent	0.5	....
Magnesium and aluminum silicates	remainder	
Alumina in silicate portion, per cent	10.0	....

(*c*) *Paste.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials, see 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

The paste shall conform to the following requirements:

	MAXIMUM	MINIMUM
Pigment, per cent	....	75.0
Linseed oil, per cent	25.0	....
Moisture and other volatile matter, per cent	0.7	....
Coarse particles and skins (total residue retained on a No. 325 sieve[1]), per cent of the dry pigment	2.5	....

Number of Tests

4. One sample shall be taken at random from each lot of 1000 packages or fraction thereof. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

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[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials, see 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

TENTATIVE SPECIFICATIONS
FOR
ZINC SULFIDE MAGNESIUM PIGMENT[1]

A.S.T.M. Designation: D 443 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover zinc sulfide magnesium pigment in the dry form, also ground in oil to form a paste.

Composition

2. (*a*) *Dry Pigment.*—The dry pigment shall consist of zinc sulfide and magnesium silicates, with or without added aluminum silicates.

(*b*) *Paste.*—The paste shall be made by thoroughly grinding the specified pigment in linseed oil.

Properties and Tests

3. (*a*) The pigment shall be similar or at least equal to a standard sample in the following qualities:

(*1*) Behavior on exposure to light,
(*2*) Ease of mixing and consistency with an approved test vehicle,
(*3*) Color and brightness,
(*4*) Tinting strength,
(*5*) Hiding power, and
(*6*) Microscopic qualities.

The standard sample and testing methods shall be mutually agreed upon by the buyer and the seller.

(*b*) *Dry Pigment.*—The dry pigment shall conform to the following requirements:

		MAXIMUM	MINIMUM
Coarse particles, per cent	total residue retained on a No. 325 sieve[2].	3.0	....
	total residue retained on a No. 100 sieve[2].	0.1	....
Zinc sulfide, per cent		....	45.0
Zinc oxide, per cent		1.0	....
Moisture, per cent		0.5	....
Magnesium and aluminum silicates		remainder	
Alumina in the silicate portion, per cent		10.0	....

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials, see 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

(*c*) *Paste.*—The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances.

The paste shall conform to the following requirements:

		Maximum	Minimum
Pigment, per cent		....	75.0
Linseed oil, per cent		25.0	....
Moisture and other volatile matter, per cent		0.8	....
Coarse particles and skins, per cent of the dry pigment	total residue retained on a No. 325 sieve[1].	3.5	....
	total residue retained on a No. 100 sieve[1].	0.2	....

Number of Tests

4. One sample shall be taken at random from each lot of 1000 packages or fraction thereof. If the packages are of such size that 1000 packages amount to more than a carload, one sample shall be taken at random from each carload.

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[1] For determining coarse particles, sieves 3 in. in diameter are recommended, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials, see 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

TENTATIVE METHOD OF TEST

FOR

RELATIVE DRY HIDING POWER OF WHITE PIGMENTS[1]

A.S.T.M. Designation: D 406 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1935; REVISED, 1937.

Scope

1. This method is intended for determining the relative dry hiding power of a white pigment and is only to be used for a comparison of standard and sample of the same type of white pigment. An optional procedure is included which permits a quantitative determination of the relative dry hiding powers of two pigments of the same type.

Definition

2. Relative dry hiding power of a white pigment is the ability of that pigment to impart hiding power to a paint prepared in the manner specified below. It is quantitatively expressed as the relative dry hiding power of this paint in terms of a similar paint prepared from a standard pigment.

NOTE.—See the definition of relative dry hiding power of a paint as given in the Tentative Method of Test for Relative Dry Hiding Power of Paints (A.S.T.M. Designation: D 344 – 37 T) of the American Society for Testing Materials.[2]

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Balance.*—Regulation laboratory balance with capacity of 100 g. and sensitivity of 0.1 g.

(*b*) *Syringe.*—Syringe of 10-ml. capacity.

(*c*) *Paint Brush.*—A good grade paint brush, 1½ in. in width.

(*d*) *Container.*—A ½-pt. can or a 250-ml. beaker.

(*e*) *Test Surface.*—A smooth-surfaced paper chart having adjacent black and white areas and coated with a suitable varnish or lacquer so as to render the surface impervious and resistant to paint liquids. The black areas of the board shall have a diffuse reflection of less than 1 per cent. The white areas shall have a diffuse reflection of 75 per cent or greater. The white areas of the boards used for the standard paint and the sample shall not differ in brightness by more than 1 per cent. The chart shall have a test area of not less than 1 sq. ft. and any suitable regular geometrical design of the contrasting areas may be used.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] See p. 866.

(*f*) *Illumination.*—A reasonable intensity of diffused light, preferably northern skylight or a source approximating northern skylight.

(*g*) *Burette.*—A standard 25-ml. burette, graduated in 0.1 ml. (stopcock controlled). The tip of the burette shall be ground optically flat.

(*h*) *Glass Muller.*—Standard type glass muller with a grinding face of from $2\frac{3}{4}$ to 3 in. in diameter. The face shall be free from blowholes and other imperfections, and kept sharp by slightly grinding with No. 303 optical emery or its equivalent and turpentine. The edge should be beveled.

(*i*) *Rubbing Surface.*—Lithographers' stone or ground plate glass slab, the surface of which is kept sharp by slightly grinding with No. 303 optical emery or its equivalent and turpentine.

(*j*) *Spatula.*—A flexible spatula having a 6-in. blade for mixing the oil and pigment.

Materials

4. The following materials will be required:

(*a*) *Standard Pigment.*—A standard white pigment of the same type as that to be tested mutually agreed upon by the purchaser and the seller.

(*b*) *Vehicle.*—The vehicle shall be prepared from the following two types of linseed oil, combining 50 per cent of each by weight to which shall be added 1 ml. of a cobalt naphthenate drier (metal content approximately 6 per cent) to each 100 g.

(*1*) Heat-bodied, alkaline, refined linseed oil, having a viscosity of E to F on the Gardner-Holdt scale[1] and an acid number of 1 to 5.

(*2*) Light, boiled linseed oil, having a viscosity of B on the Gardner-Holdt scale,[1] an acid number of 2 to 5, and color not darker than No. 11 on the 1933 Gardner Color Standard.[2]

Procedure

5. The relative dry hiding power of the white pigment shall be determined as follows:

(*a*) The standard white pigment and vehicle shall be first mulled to a stiff paste and then reduced to a paint of reasonably good brushing properties. Quantities of pigment and vehicle for both operations shall be mutually agreed upon by the purchaser and the seller. The quantities of pigment and vehicle used should produce paints of reasonably good brushing properties from both standard and sample. The following table is intended to serve only as a guide:

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[1] Gardner's Laboratory Manual, Seventh Edition, October, 1935.

[2] Gardner's Laboratory Manual, Sixth Edition, October, 1933, p. 161.

SUGGESTED QUANTITIES OF PIGMENT AND VEHICLE.

PIGMENT	PIGMENT, G.	OIL FOR MULLING, ML.	OIL FOR REDUCING SINGLE MULLING, ML.
Zinc oxide	17.8	5.6	6.3
Leaded zinc oxide	16.3	[illegible]	8.7
Antimony oxide	25.3	5.3	5.3
Basic carbonate white lead	25.5	4.3	7.0
Basic carbonate white lead (high oil)	25.5	6.2	6.5
Basic sulfate white lead	25.5	5.0	6.0
Titanium dioxide	11.6	6.6	5.6
Titanated lithopone	17.9	7.5	3.4
Titanium barium (30 per cent)	18.9	5.9	4.6
Titanium calcium (low oil)	13.9	6.1	4.4
Titanium calcium (high oil)	12.4	8.1	3.0
Titanium magnesium	12.9	7.6	3.8
Lithopone (low-oil absorption)	13.9	3.8	8.0
Lithopone (high-oil absorption)	10.5	4.4	8.2
Zinc sulfide barium	11.8	4.2	8.0
Zinc sulfide magnesium	10.6	5.0	6.9
Zinc sulfide	10.7	4.4	7.9

(*b*) The amount of standard white pigment shall be weighed accurately and the corresponding quantity of vehicle measured out, taking care to allow sufficient time for complete drainage of the burette (approximately 5 min.). The materials shall be placed on the rubbing slab and worked into a paste with the spatula. Then the paste shall be rubbed up with the glass muller, using a stroke approximately 6 in. in width and from 12 to 15 in. in length. In counting rubs, one stroke up and one stroke back shall be considered to be one rub. The muller shall be allowed to travel up one side and back the other side, twisting the muller slightly at the top and bottom of each half stroke to help work in the pigment After each 25 rubs with the muller, the pigment shall be "picked up" with the spatula by scraping the face of the muller and gathering the paste on the slab into a daub. The mulling shall be continued until the paste is given 100 rubs. This paste shall then be thinned with the additional vehicle and transferred to the container.

(*c*) The sample of white pigment to be tested shall be treated in the same manner as the standard, as described in Paragraphs (*a*) and (*b*).

(*d*) Brushouts of these paints shall be prepared as described in Paragraph (*e*) at a reasonable spreading rate mutually agreed upon by the purchaser and the seller.

(*e*) The paint prepared from the standard pigment shall be well stirred and the brush dipped into the paint and worked out on a suitable surface. The desired quantity of paint shall be taken up in the syringe, and the syringe and wet brush weighed in the empty container. The paint shall then be spread over the surface of the chart, the syringe returned to the empty container, and the paint brushed out uniformly over the test surface, care being taken to cover only the test area. The empty syringe, wet brush, and container shall again be weighed. The loss of weight represents the weight of paint applied to the test surface. A test chart shall then be prepared from the paint made with the sample pigment in the same manner and at the same spreading rate. Both painted test surfaces shall then be set aside to dry.

(*f*) After the test surfaces are thoroughly dry, they shall be placed side by side against a flat surface. The standard and sample brushouts shall be viewed in juxtaposition under a reasonable intensity of diffused light, preferably northern skylight or a source approximating northern skylight. The observer should view the brushouts from a distance of 5 to 10 ft.

Relative Dry Hiding Power

6. If the apparent contrast existing on the brushout of the sample of white pigment is adjudged equal to or less than that existing on the brushout of the standard white pigment, the sample shall be said to meet the standard pigment in relative dry hiding power as defined in Section 2.

Optional Procedure

7. (*a*) In many cases it is not sufficient to say that the sample pigment is better or poorer than the standard, and the amount of difference must be determined quantitatively. The optional procedure described in the following Paragraphs (*b*) and (*c*) is recommended for determining quantitative differences in hiding power. It may be used in place of the procedure outlined in Section 5.

(*b*) The paint prepared from the sample of white pigment shall be applied to a brushout board as described in Section 5 (*e*) at the spreading rate mutually agreed upon. A series of brushouts shall be made from the paint prepared from the standard white pigment. The difference in spreading rate between successive steps in this series should be approximately 15 per cent. The range of spreading rate covered by this series should be sufficient to give contrasts above and below that of the brushout of the sample pigment (estimated from the probable difference in relative dry hiding power, if known) or should cover the entire range of spreading rate convenient to apply with the standard paint. The painted test surfaces shall be set aside to dry.

(*c*) Under the conditions of illumination and viewing, as described in Section 5 (*f*), the brushout of the sample paint shall be compared with a series of brushouts of the standard paint, numbered, beginning with one, in order of decreasing spreading rate for convenience of handling. It will be found to be equal in contrast to one brushout of the standard or to be intermediate in contrast between two adjacent standards in the series of standard brushouts. If the sample is found to be between two adjacent standards in contrast, the sample shall be viewed against these standards with the higher and lower standard on either side. For purposes of grading, the contrast interval between the two standards shall be considered as 10 arbitrary units. The position of the sample shall be estimated on this arbitrary scale. Thus, if the sample appears equally distant in contrast from either standard, it will be graded 5 on the arbitrary scale. If nearer the standard of less contrast, it will be graded 6, 7, 8, or 9 as the case might be. The grading shall be recorded in terms of the number of the standard brushout of higher spreading rate followed by a decimal point and the grading of the arbitrary scale. In making this grading it is important that the sample be graded between the two standards with the standard of lesser contrast first on the right and then on the left. The two gradings thus obtained shall be averaged to give the final grading figure.

Hiding Power Calculation

8. The hiding power of the sample of white pigment shall be calculated as follows:

$$\text{Hiding power} = \frac{(W_1 - W_2)\dfrac{X}{10} + W_2}{W_3} \times 100$$

where W_1 = the weight of paint on the brushout of the heavier grading standard,

W_2 = the weight of paint on the brushout of the lighter grading standard,

W_3 = the weight of paint on the brushout of the sample pigment, and

X = the contrast grading (decimal portion of the recorded grading).

TENTATIVE METHODS OF ROUTINE ANALYSIS

OF

ZINC YELLOW PIGMENT (ZINC CHROMATE YELLOW)[1]

A.S.T.M. Designation: D 444 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1937.

Scope

1. These methods cover the procedures for physical tests and chemical analysis of the pigment commonly known as "zinc yellow," or "zinc chromate yellow" (Notes 1, 2 and 3).

Treatment of Sample

2. Dry pigments, if lumpy or not finely ground, shall be ground to a fine powder for analysis. Large samples may be thoroughly mixed and a representative portion taken and powdered, if lumpy or not finely ground. The sample in all cases shall be thoroughly mixed before taking portions for analysis. Extracted pigments shall be ground to a fine powder, passed through a No. 80 sieve to remove any skins, and thoroughly mixed. The weighed portions of extracted pigments should be moistened with a little alcohol before adding reagents for analysis. All samples shall be preserved in stoppered bottles or containers.

METHODS OF PHYSICAL TESTING

Specific Gravity

3. True specific gravity shall be determined in accordance with the Standard Methods of Test for Specific Gravity of Pigments (A.S.T.M. Designation: D 153) of the American Society for Testing Materials.[2]

Tinting Strength

4. Tinting strength shall be determined in accordance with the Standard Methods of Test for Mass Color and Tinting Strength of Dry Color Pigments or Pastes (A.S.T.M. Designation: D 387) of the American Society for Testing Materials.[3]

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 638.

[3] *Ibid.*, p. 651.

Moisture

5. Weigh 6 g. of zinc yellow in a tared moisture dish. Dry the specimen over night at 110 C. Cool, and weigh the specimen and calculate the percentage of moisture as follows:

$$\text{Moisture, per cent} = \frac{\text{Loss of weight} \times 100}{\text{Weight of sample}}$$

Combined Water and Moisture[1]

6. Weigh 3 g. of pigment and transfer to a porcelain boat. Place the boat in a combustion tube. Connect the apparatus and heat for 4 hr. The weight increase of the absorption tube represents the "combined water," report as the percentage of combined water.

METHODS OF CHEMICAL ANALYSIS

Solution of Sample

Dissolve 4 g. of the sample in 50 ml. of cold 6 *N* H_2SO_4 (160 ml. concentrated H_2SO_4 per liter of solution). It should dissolve completely at this stage. Transfer the solution to a 500-ml. calibrated flask and make up to 500 ml. The concentration of the solution should be approximately 0.1 *N* chromic acid.

Chromium

7. (*a*) *Special Solution Required.*—Standard sodium thiosulfate solution (0.1 *N*).

(*b*) *Procedure.*—To a 400-ml. beaker containing 200 ml. of water, add 2 g. of potassium iodide (solid) and 5 ml. of H_2SO_4 (sp. gr. 1.42). (Final acidity should be approximately 5 ml. of acid per 100 ml. of solution). Pipette 25 ml. of the chromic acid solution into the solution of KI and H_2SO_4 and after 5 min. titrate with the 0.1 *N* standard sodium thiosulfate solution, using starch as an internal indicator. The percentage of chromic acid may be calculated from the following values:

1 ml. of *N* sodium thiosulfate = 0.01733 g. of Cr. or 0.03334 g. of CrO_3.

Report the amount calculated as percentage of CrO_3.

Zinc

8. (*a*) *Special Solution Required.*—Potassium ferrocyanide solution. The $K_4Fe(CN)_6$ solution is standardized as follows: Dissolve 42.5 g. of $K_4Fe(CN)_6$ per liter of solution. Dissolve 0.4 g. pure zinc in 25 ml. of concentrated HCl. After the zinc has completely dissolved, dilute to 300 ml. Neutralize the solution with NH_4OH, add 2 g. NH_4Cl and 3 ml. of HCl (sp. gr. 1.19). Titrate the solution with $K_4Fe(CN)_6$ using uranyl acetate as an external indicator on a spot tile and ferrous ammonium sulfate

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[1] Refer to Fox and Bowles, "Analysis of Pigments, Paints and Varnishes," p. 57.

as an internal indicator. Calculate the strength of the potassium ferrocyanide solution in terms of grams of zinc as follows:

$$1 \text{ ml. of } K_4Fe(CN)_6 = \frac{\text{Grams of zinc}}{\text{Milliliters of } K_4Fe(CN)_6}$$

(*b*) *Procedure.*—Nearly neutralize 100 ml. of the solution of the sample with NH_4OH, add methyl orange indicator and continue neutralization with weak NH_4OH (1:10) until exactly neutral. There should be no precipitate formed. The solution must be barely acid before beginning the precipitation and the acidity (due to liberated H_2SO_4) should not increase to more than 0.1 *N*. The precipitation of zinc sulfide ceases if the acidity increases to more than 550 mg. of H_2SO_4 per 100 ml. of solution; below 0.1 *N* acid concentration, the precipitation is complete. Pass H_2S through the solution at a rapid rate (at least eight bubbles per second) for at least 40 min., keeping the temperature below 50 C. Filter the solution and wash the precipitate with water.

Dissolve the ZnS precipitate in hot diluted HCl (1:3). Wash the filter paper well with hot water. Boil the H_2S out of the ZnS solution, neutralize with NH_4OH and adjust the volume to 300 ml. Add 2 g. of NH_4Cl, and 3 ml. of HCl (sp. gr. 1.19), and heat to boiling. Titrate the hot solution with standard potassium ferrocyanide solution using uranyl acetate as an external indicator on a spot plate, and 1 to 2 drops of ferrous ammonium sulfate as an internal indicator. The percentage of zinc in the specimen may be calculated from the following formula:

$$\text{Zinc, per cent} = \frac{\text{Milliliters of } K_4Fe(CN)_6 \times \text{grams of zinc per milliliter} \times 100}{\text{Weight of sample}}$$

$$\text{Zinc oxide, per cent} = \text{percentage of zinc} \times 1.2447$$

Report the amount calculated as percentage of ZnO.

Total Alkali Metals

9. Dissolve 1 g. of zinc yellow in 10 ml. of acetic acid (1:1), add 25 ml. of water and heat until completely dissolved. Dilute to approximately 250 ml. and heat the solution to boiling. Add 20 ml. of lead acetate (10 per cent) and allow to settle. Filter off the precipitate and wash well with hot water. Saturate the filtrate with H_2S, observing the precautions as outlined in the procedure for determining zinc (Section 8 (*b*)); zinc and excess lead will be precipitated. (It is necessary to keep down the concentration of acetic acid to ensure complete removal of the zinc.) Filter and wash the precipitate with cold water. Boil the filtrate to expel H_2S, then add 3 ml. of H_2SO_4 and boil down to a small volume. Transfer the concentrate to a weighed silica dish and evaporate to dryness on a hot plate. Heat to complete dryness over a bunsen burner. Add solid $(NH_4)_2CO_3$ to

the residue and heat till volatilized. Repeat the operation four times, cool the dish, and weigh. Add a few crystals of $(NH_4)_2CO_3$, and again heat the residue until volatilized. Cool, and weigh the dish and repeat the operation until constant weight is obtained.

The percentage of total alkali may be calculated as follows:

$$\text{Total sulfate } (Na_2SO_4 \text{ and } K_2SO_4), \text{ per cent} = \frac{\text{Weight of alkaline sulfates} \times 100}{\text{Weight of sample}}$$

$$\text{Total Alkali } (K_2O), \text{ per cent} = \text{percentage of } K_2SO_4 \times 0.541$$

Report the amount calculated as percentage of K_2O.

NOTE.—The above determination would include both potassium and sodium if present. Flame test for sodium should be made at this point, and if present the procedure for sodium should be followed (see Section 12 (*a*) and (*b*)).

Sulfates

10. Dissolve 1 g. of zinc yellow in 15 ml. of HCl (sp. gr. 1.19). Heat the solution to boiling and add 5 ml. of alcohol by drops to insure the reduction of chromium. Boil the solution down to a small volume. Dilute the concentrate to 300 ml. with water and neutralize with Na_2CO_3 adding 3.5 g. in excess. Heat the solution to boiling and boil to ensure the conversion of the insoluble sulfates to the soluble form. Allow the precipitate in the solution to settle on a hot plate and filter. Wash the precipitate with hot water. Neutralize the filtrate with HCl and boil to expel the CO_2. Adjust the acidity to 2.5 per cent with HCl and add drop-by-drop 15 ml. of barium chloride (20 per cent) to the solution, keeping the latter boiling. Allow the precipitate to settle on a hot plate, filter through a weighed Gooch crucible, wash, dry, ignite, cool, and weigh. The increase in weight represents the barium sulfate.

$$\text{Sulfate, per cent} = \frac{\text{Weight } BaSO_4 \times 0.343 \times 100}{\text{Weight of sample}}$$

Report the amount calculated as percentage of SO_3.

Chlorine

11. Dissolve 1 g. of zinc yellow in 20 ml. of H_2SO_4 (2:3) and heat the solution to boiling. Add drop-by-drop 5 ml. of alcohol to the boiling solution; reduction of the chromium is complete when a clear green solution is obtained. Dilute the solution to approximately 250 ml., neutralize with Na_2CO_3 and add a slight excess. Boil the solution a few minutes, filter, and wash. Acidify with diluted HNO_3 (1:1), adding 25 ml. in excess. Add 10 ml. of standard silver nitrate solution (0.05 *N*) and titrate the excess silver nitrate with standard potassium sulfocyanate (0.05 *N*), using ferric sulfate as an internal indicator. A red tinge in the solution indicates the end point. The percentage of chlorine in the sample may be calculated as follows:

$$\text{Chlorine, per cent} = \frac{\text{milliliters of AgNO}_3 - (\text{milliliters of KCNS} \times \text{factor}) \times \text{grams of Cl per milliliter of AgNO}_3 \times 100}{\text{Weight of sample}}$$

Report the amount calculated as percentage of chlorine.

Sodium

12. (*a*) *Special Solutions Required.*—Double acetate solution, prepared as follows:

Solution A	Uranyl acetate	10 g.
	Acetic acid (30 per cent)	6 g.
	Water	65 g.
Solution B	Zinc acetate	30 g.
	Acetic acid (30 per cent)	3 g.
	Water	65 g.

When the above solutions A and B are each completely dissolved, mix them, and allow to cool to 20 C. ± 1 C. Keep the resulting solution at this temperature for several hours. Filter off the triple salt which crystallizes out and which may be assumed to be saturated with respect to sodium at 20 C. since the reagent usually contains sufficient sodium to give a precipitate.

(*b*) *Procedure.*—Proceed as in the determination of total alkali (Section 9) to the weighed sulfates. Dissolve the sulfates in 10 to 15 ml. of water. Add to the solution 100 ml. of zinc-uranyl acetate reagent and allow to stand at 20 C. ± 1 C. for 30 to 45 min. stirring occasionally. (Scratch the side of the beaker occasionally to start crystallization.) Filter the solution through a tared Gooch crucible and wash with five successive amounts of 2 ml. of zinc-uranyl acetate reagent, then with five 2-ml. portions of alcohol (previously saturated with sodium zinc-uranyl acetate) and finally with ether. Dry at 40 C. for 10 to 15 min., and weigh. The percentage of sodium may be calculated as follows:

$$\text{Sodium, per cent} = \frac{\text{Weight of precipitate} \times 0.01495 \times 100}{\text{Weight of sample}}.$$

NOTE.—The precipitate is slightly soluble in water, insoluble in the precipitant, alcohol, and ether. Barium, calcium, magnesium, and zinc do not interfere with the precipitation. Potassium has little effect if less than 0.05 g. is present; lithium, phosphates, oxalates, and tartrates should be absent.

Calcium

13. Proceed as in the determination of sulfates (Section 10) to the acidification of the sodium carbonate solution. Make the solution ammoniacal and then slightly acid with acetic acid. Add 15 ml. of saturated ammonium acetate solution and again make ammoniacal with NH_4OH and allow to stand on a hot plate over night. Filter off any precipitate in the morning. Wash free from oxalates with NH_4OH (10 per cent). Dissolve the oxalate precipitate in hot diluted H_2SO_4. Dilute the solution to approx-

imately 300 ml. and add 10 ml. of H_2SO_4. Heat the solution to boiling and titrate with 0.1 N potassium permanganate solution.

$$\text{Calcium, per cent} = \frac{\text{Milliliters of } KMnO_4 \times N \times 0.20}{\text{Weight of sample}} \times 100$$

where N = the normality of the $KMnO_4$ solution.

Ammonia[1]

14. Distill 1 g. of the zinc yellow pigment with NaOH solution expelling the ammonia and collecting it in a known excess of 0.1 N H_2SO_4. Titrate the excess H_2SO_4 with 0.1 N NaOH.

EXPLANATORY NOTES

NOTE 1.—According to the work of Denslow, it appears probable that zinc yellow is a definite salt, the "oxide formula" of which would be $K_2O \cdot 4ZnO \cdot 4CrO_3 \cdot 3H_2O$.

NOTE 2.—For ordinary routine work, the following determinations should be sufficient: combined water, chromium (CrO_3), zinc (ZnO), total alkali metals (K_2O) and sulfates (SO_3). These should give a summation close to 100 per cent.

NOTE 3.—In commercial "zinc yellow" pigments, using the above described methods, the percentage of zinc calculated as ZnO generally varies between 36.9 and 37.8. The chromium as CrO_3 generally varies between 41.3 and 43.6 per cent. The sulfates as SO_3 generally vary between 1.4 and 3.3 per cent. The "combined water" generally varies between 6.1 and 6.7 per cent. The alkali metals expressed as K_2O generally vary between 10.4 and 11.8 per cent. One sample showed 0.17 per cent moisture and 0.51 per cent sodium expressed as Na_2O.

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[1] This procedure is occasionally but not usually employed for preparing zinc yellow pigments.

TENTATIVE METHOD OF ROUTINE ANALYSIS

OF

DRY CUPROUS OXIDE[1]

A.S.T.M. Designation: D 283 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. This method covers the chemical analysis of the pigment, dry cuprous oxide.

Treatment of Sample

2. Dry pigments, if lumpy or not finely ground, shall be ground to a fine powder and thoroughly mixed (Note 1). Large samples may be thoroughly mixed and a representative portion taken and powdered, if lumpy or not finely ground. The sample in all cases shall be thoroughly mixed before taking portions for analysis. All samples shall be preserved in stoppered bottles or containers (Note 2).

TOTAL COPPER

Total Copper

3. Weigh accurately a dry sample of from 0.15 to 0.2 g. of the material, transfer it to a 250-ml. beaker, add 10 to 15 ml. of HCl (sp. gr. 1.19) and 1 to 2 ml. of HNO_3 (sp. gr. 1.42) and boil for several minutes. Add 10 ml. of H_2SO_4 (sp. gr. 1.84) and heat until copious fumes of sulfuric acid are evolved. Cool, add cautiously while stirring, 50 ml. of cold water, and boil to dissolve the sulfates. Cool, filter off any insoluble matter (SiO_2, $PbSO_4$, etc.) and wash the filter and residue until free from copper, with a cold H_2SO_4 solution (1 to 2 per cent), catching the filtrate and washings in a 250-ml. beaker, keeping the total volume of the solution down to about 150 ml. Place in the solution a piece of clean, pure sheet aluminum having a minimum purity of 99.80 per cent and to contain a maximum of 0.02 per cent copper. The sheet aluminum is convenient to use in the form of a cylinder about 3.5 to 4 cm. in diameter by 3.5 to 4 cm. in height, with a

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.
This method is in effect a tentative revision of and is intended to supersede when adopted the present Standard Method of Routine Analysis of Dry Cuprous Oxide (A.S.T.M. Designation: D 283 – 36), 1936 Book of A.S.T.M. Standards, Part II, p. 624.

small extending piece about 7 to 8 cm. in height as a means of removing the cylinder. Cover the beaker and heat to gentle boiling for about 30 min. or until the copper is completely precipitated as a metallic sponge on the aluminum. Thoroughly wash the copper sponge from the aluminum into a 250-ml. beaker and carefully examine the aluminum sheet for any remaining particles of copper. Wash the copper sponge several times by decantation with hot water, filtering the wash water through a 9-cm. filter paper, but retaining the copper as completely as possible in the beaker. The filtrate and washings may be discarded or used for the determination of iron, if present (Note 4). Add from 10 to 15 ml. of ferric chloride-hydrochloric acid solution (prepared by dissolving 250 g. of $FeCl_3 \cdot 6H_2O$ in 500 ml. of HCl (1:1)), on the filter paper, catching this in the beaker containing the copper sponge. The copper generally dissolves in a few minutes without the need of heating. Break up any large particles of the copper sponge with a stirring rod. When the copper is completely dissolved, wash the filter thoroughly with cold water and finally, dilute the filtrate to about 200 ml. with cold water, add 5 ml. of syrupy phosphoric acid and titrate the cold solution at once with 0.1 N $KMnO_4$ solution. Run a blank on the reagents used, and calculate the final corrected figure to percentage of total copper (1 ml. of 0.1 N $KMnO_4$ is equivalent to 0.0032 g. Cu).

METALLIC COPPER AND CUPROUS OXIDE[1]

Apparatus

4. *Filter.*[2]—The filter consists of a 55-mm. filter tube of the type used for small Gooch crucibles. In the opening is placed a perforated porcelain button which is seated at right angles to the stem. An asbestos pad is built up over the button and securely set by tamping with a glass rod. The filter must be packed in such a way that the tube may be held in an inverted position without dislodging either the disk or the pad. It has been found that a tube about 60 by 25 mm., with a stem 110 mm. in length, is a convenient size. A 20-mm. Gooch crucible button serves to support the asbestos pad.

Special Solutions Required

5. (*a*) *Extraction Solution.*—Dissolve 6 g. of c. p. hydrazine sulfate in 1 liter of NH_4OH (sp. gr. 0.90).

(*b*) *Ferric Chloride Solution.*—Add 150 g. of $FeCl_3 . 6H_2O$ and 300 ml. of HCl (sp. gr. 1.19) to 800 ml. of air-free, carbon-dioxide-saturated water.

(*c*) *Indicator.*—Shake 2 g. of barium diphenylamine sulfonate,[3] 50 ml. of carbon-dioxide-saturated distilled water, and 5 g. of sodium sulfate until a uniform suspension of barium sulfate is obtained. Dilute the suspension with 50 ml. of water and allow to settle. When clear, decant the supernatant liquid through a filter and preserve in a dark bottle.

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[1] L. C. Hurd and A. R. Clark, "Determination of Metallic Copper in Cuprous Oxide - Cupric Oxide Mixture," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 8, No. 5, p. 380 (1936).

[2] For further information on the filter see W. Geilman, and F. Z. Weibke, *Zeitschrift für anorganische und allgemeine Chemie*, Vol. 199, p. 120 (1931).

[3] Barium diphenylamine sulfonate may be obtained from the Eastman Kodak Co.

(*d*) *Phosphoric Acid.*—Prepare a solution of ortho-phosphoric acid, 85 per cent reagent, conforming to specifications of the American Chemical Society.

(*e*) *Potassium Bichromate Solution* (0.1 *N*).—Prepare a 0.1 *N* solution by dissolving 4.903 g. of reagent quality potassium bichromate in water, diluting to 1 liter.

(*f*) *Water.*—Boil vigorously distilled water for several minutes and allow to cool in an atmosphere of carbon dioxide. Allow a slow current of the gas to pass continuously through the storage bottle.

Metallic Copper

6. (*a*) Weigh accurately a sample of suitable size on a small watch-glass, and place the glass and contents in a dry, wide-mouth 250-ml. Erlenmeyer flask. If the material is an electrolytic product low in copper, it is advisable to take a 1 to 2-g. sample. If it is from a "thermal process," the percentage of copper is usually large and a smaller sample will suffice. Displace the air in the flask with carbon dioxide (15 to 20 cu. ft. per hr.) and add 10 ml. of ethyl alcohol to dissolve any oil present in the sample. Without interrupting the flow of gas, add 150 ml. of extraction solution (Note 5). Break up any lumps of oxide with a stirring rod. Violent agitation should be avoided. The carbon dioxide inlet should be about 5 cm. (2 in.) above the surface of the liquid (Note 6). The time required for complete solution of all cuprous oxide varies between 1 and 5 min. depending upon the amount and character of the sample under investigation.

(*b*) When the cuprous oxide has completely dissolved, as evidenced by the total disappearance of red particles, connect the filter to a suction and slowly lower it into the flask. As soon as the bulk of the solution is removed, rinse the flask with carbon-dioxide-saturated water and continue the filtration and washing. Five of six 100-ml. portions of wash water will suffice to remove all the original extraction solution containing the dissolved cuprous oxide. Then disconnect the filter and push the pad and contents back into the flask with a glass rod. Add 15 ml. of ferric chloride solution and warm the flask to dissolve the cupric oxide - copper residue. When all particles have disappeared, cool the solution to below 40 C. and add 10 ml. of phosphoric acid and 3 drops of indicator. Titrate the solution by running the bichromate solution into the flask. The endpoint is evidenced by a change from pea green to an intense purple color.

(*c*) *Calculation.*—Calculate the percentage of copper present (Note 7) from the following formula:

$$\text{Copper, per cent} = \frac{\text{Normality of } K_2Cr_2O_7 \times \text{milliliters of } K_2Cr_2O_7 \times 0.03179}{\text{Weight of sample in grams}} \times 100$$

Cuprous Oxide

7. Dissolve a 0.2-g. sample in ferric chloride and titrate the ferrous iron produced with potassium bichromate in the manner described. It is essential that the solution of the sample be carried out in an atmosphere of carbon dioxide or other inert gas. From the volume of bichromate solution equivalent to both cuprous oxide and copper, calculate the true percentage of cuprous oxide in the sample according to the following formulas:

Let

$$\frac{\text{ml.}_1}{\text{w}_1} = \text{milliliters required for copper in 1 g. of sample, and}$$

$$\frac{\text{ml.}_2}{\text{w}_2} = \text{milliliters required for copper plus cuprous oxide in 1 g. of sample,}$$

Then

$$\frac{\text{ml.}_2}{\text{w}_2} - \frac{\text{ml.}_1}{\text{w}_1} = \text{milliliters required for cuprous oxide in 1 g. of sample, and}$$

Therefore

$$\text{Cuprous oxide, per cent} = \left(\frac{\text{ml.}_2}{\text{w}_2} - \frac{\text{ml.}_1}{\text{w}_1}\right) 0.07157 \times N \text{ of } K_2Cr_2O_7 \times 100$$

but

$$\frac{\text{ml.}_1}{\text{w}_1} = \frac{\text{Percentage of copper}}{N \text{ of } K_2Cr_2O_7 \times 0.03179 \times 100}$$

Therefore

$$\begin{array}{c}\text{Cuprous oxide,}\\ \text{per cent}\end{array} = \left(\frac{\text{ml.}_2}{\text{w}_2} - \frac{\text{Percentage of copper}}{N \text{ of } K_2Cr_2O_7 \times 0.03179 \times 100}\right) \times \begin{array}{c}0.07157 \times N \text{ of}\\ K_2Cr_2O_7 \times 100\end{array}$$

The calculation of the percentage of cuprous oxide is simplified if the sample taken for the copper determination be a simple multiple of that in the oxide analysis. In actual practice it has been found that a 1.000-g. sample for metallic copper and a 0.2000-g. sample for cuprous oxide are of convenient size. In this case one-fifth of the volume of bichromate used in the determination of metallic copper is subtracted from the total titration of the smaller sample to give the volume of bichromate actually equivalent to the cuprous oxide in the sample.

Report

8. Report the metallic copper (Cu) and the cuprous oxide (Cu_2O). Subtract the copper equivalents from the percentage of total copper as determined in Section 2, calculate the remainder to cupric oxide and report as percentage of CuO (Cu × 1.252 = CuO).

EXPLANATORY NOTES

NOTE 1.—It is very important that the sample be thoroughly mixed. Some samples of cuprous oxide are not homogeneous, and for this reason are likely to give trouble when an attempt is made to obtain concordant results. By placing a few grams of a sample on a sheet of white paper, and drawing it out with a spatula, it is frequently found that the sample contains coarse particles of black scale, along with small balls of bright red cuprous oxide. Thus it may be necessary to grind such a product thoroughly in a mortar and pestle, in order to get it homogeneous. It is possible that during this operation a slight oxidation may take place.

NOTE 2.—Caution should be used in protecting the sample from oxidation.

NOTE 3.—A microscopic examination is helpful in showing the presence of metallic Cu, red Cu_2O, and CuO. Washing some of the sample through a fine sieve (No. 325) may reveal the presence of metallic copper retained on the sieve.

NOTE 4.—Commercial cuprous oxides may contain moisture and small amounts of iron, chlorine, oil, and acid insoluble matter.

NOTE 5.—If the ammonium hydroxide-hydrazine sulfate solution is allowed to stand in contact with the samples for more than 10 min., the loss in copper may amount to more than 2 mg.

NOTE 6.—Care must be taken not to allow the extraction solution to become saturated with carbon dioxide. The gas inlet to the flask should not be placed beneath the surface of the liquid, to avoid solubility errors.

NOTE 7.—Commercial samples of cuprous oxide have shown from 0.22 to 11.8 per cent Cu.

TENTATIVE SPECIFICATIONS
FOR
RAW SOYBEAN OIL[1]

A.S.T.M. Designation: D 124 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1922; REVISED, 1933, 1937.

Properties

1. Raw soybean oil shall conform to the following requirements:

	MAXIMUM	MINIMUM
Foots, per cent	2.5	...
Loss on heating at 105 to 110 C., per cent	0.2	...
Specific gravity at 15.5/15.5 C	...	0.924
Acid number	3.0	...
Saponification number	...	190
Unsaponifiable matter, per cent	1.5	...
Appearance	clear and transparent at 65 C.	
Iodine number (Wijs)	...	131
Color	Not darker than a freshly prepared solution of 1.0 g. potassium bichromate in 100 ml. pure H_2SO_4 (sp. gr. 1.84) or its equivalent in iron-cobalt solution or in Lovibond glass.	

METHODS OF TESTING

Solutions Required

2. The following reagents will be required:

(*a*) *Acetone.*—Acetone shall conform to the Standard Specifications for Acetone (A.S.T.M. Designation: D 329) of the American Society for Testing Materials.[2]

(*b*) *Acid Calcium Chloride Solution.*—Saturate with calcium chloride a mixture of 90 parts of water and 10 parts of HCl (sp. gr. 1.19).

(*c*) *Standard Sodium Thiosulfate Solution.*—Dissolve pure sodium thiosulfate in distilled water that has been well boiled to free it from carbon dioxide in the proportion so that 24.83 g. crystallized sodium thiosulfate will be present in 1000 ml. of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine. (See Treadwell-Hall, *Analytical Chemistry*, Vol. II, sixth edition, pp. 551 and 553.) This solution will be approximately 0.1 *N* and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly 0.1 *N* strength. Preserve in a stock bottle with a guard tube filled with soda lime.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 784.

(*d*) *Starch Solution.*—Stir up 2 to 3 g. of potato starch or 5 g. of soluble starch with 100 ml. of 1 per cent salicylic acid solution, add 300 to 400 ml. boiling water, and boil the mixture until the starch is practically dissolved. Dilute to 1 liter.

(*e*) *Potassium Iodide Solution (15 per cent).*—Dissolve 150 g. of potassium iodide free from iodate in distilled water and dilute to 1000 ml.

(*f*) *Wijs Iodine Monochloride Solution.*—Dissolve iodine in glacial acetic acid that has a melting point of 14.7 to 15 C. and which is free from reducing impurities in the proportion so that 13 g. of iodine will be present in 1000 ml. of solution. The preparation of the iodine monochloride solution presents no great difficulty, but it shall be done with care and accuracy in order to obtain satisfactory results. There shall be in the solution no sensible excess either of iodine or more particularly of chlorine over that required to form the monochloride. This condition is most satisfactorily attained by dissolving in the whole of the acetic acid to be used the requisite quantity of iodine, using a gentle heat to assist the solution, if it is found necessary. The halogen content can be accurately measured by titration of a portion of the solution with 0.1 *N* sodium thiosulfate using the potassium iodide solution (15 per cent) and the starch indicator as described in Section 3 (*g*) in the method for the determination of the iodine number. Set aside a small portion of this solution while pure, and pass dry chlorine into the remainder until the halogen content of the solution is doubled. Ordinarily, it will be found that by passing the chlorine into the main part of the solution until the characteristic color of free iodine has just been discharged, there will be a slight excess of chlorine, which is corrected by the addition of the requisite amount of the unchlorinated portion until all free chlorine has been destroyed. A slight excess of iodine does little or no harm, but excess of chlorine must be avoided.

(*g*) *Standard Sodium Hydroxide Solution.*—Prepare a stock concentrated solution of sodium hydroxide by dissolving NaOH in water in the proportion of 200 g. NaOH to 200 ml. water. Allow this solution to cool and settle in a stoppered bottle for several days. Decant the clear liquid from the precipitate of sodium carbonate into another clean bottle. Add clear barium hydroxide solution until no further precipitate forms. Again allow to settle until clear. Draw off about 175 ml. and dilute to 10 liters with freshly boiled distilled water. Preserve in a stock bottle provided with a large guard tube filled with soda lime. Determine the exact strength by titrating against pure benzoic acid (C_6H_5COOH) using phenolphthalein as indicator. (See Bureau of Standards *Scientific Paper No. 183.*) This solution will be approximately 0.25 N, but do not attempt to adjust it to any exact value. Determine its exact strength and make proper corrections in using it.

(*h*) *Alcoholic Sodium Hydroxide Solution.*—Dissolve pure NaOH in 95 per cent ethyl alcohol in the proportion of about 22 g. per 1000 ml. Let stand in a stoppered bottle. Decant the clear liquid into another bottle and keep well stoppered. This solution should be colorless or only slightly yellow when used; it will keep colorless longer if the alcohol is previously treated with NaOH (about 80 g. to 1000 ml.), kept at about 50 C. for 15 days, and then distilled. For an alternate method see *Journal*, American Chemical Society, 1906, p. 395.

(*i*) *Half Normal Sulfuric Acid Solution.*—Add about 15 ml. of H_2SO_4 (sp. gr. 1.84) to distilled water, cool and dilute to 1000 ml. Determine the exact strength by titrating against freshly standardized NaOH or by any other accurate method. Either adjust to exactly 0.5 N strength or leave as originally made, applying appropriate correction.

Methods of Testing

3. The oil shall be tested in accordance with the following methods:

(*a*) *General.*—The laboratory sample shall be thoroughly mixed by shaking, stirring, or pouring from one vessel to another and the samples for the individual tests taken from this thoroughly mixed sample.

(*b*) *Foots.*—With all materials at a temperature between 20 and 27 C., mix by shaking for exactly one minute in a graduated tube, 25 ml. of the well-shaken sample of oil, 25 ml. of acetone and 10 ml. of the acid calcium chloride solution. Clamp the tube in a vertical position where settling may take place for 24 hr. The temperature during this period should be between 20 and 27 C.

At the end of the 24 hr., determine the volume of the stratum lying between the clear calcium chloride solution and the clear acetone and oil mixture to the nearest 0.1 ml. or fraction thereof. This volume multiplied by four expresses the amount of foots present as a percentage by volume.

The graduated tube referred to may be a burette or a color comparison tube. It should have an internal diameter of from 1.0 to 1.5 cm., and a capacity of not less than 70 ml. The graduations in 0.1 ml. should extend at least from 10 ml. to 50 ml. above the bottom of the tube.

(*c*) *Loss on Heating at 105 to 110 C.*—Place 10 g. of the oil in an accurately weighed 200-ml. Erlenmeyer flask; weigh. Heat in an oven at a temperature between 105 and 110 C. for 30 minutes; cool and weigh. Calculate the percentage loss. This determination shall be made in a current of dry carbon dioxide gas.

(*d*) *Specific Gravity.*—Use a pycnometer accurately standardized and having a capacity of at least 25 ml., or any other equally accurate method, making the test at 15.5 C., water being 1 at 15.5 C.

(*e*) *Acid Number.*—Weigh from 5 to 10 g. of the oil. Transfer to a 300-ml. Erlenmeyer flask. Add 50 ml. of neutral 95 per cent ethyl alcohol. Put a condenser loop inside the neck of the flask. Heat on a steam bath for 30 minutes. Cool and add phenolphthalein indicator. Titrate to a faint permanent pink color with the 0.2 N sodium hydroxide solution. Calculate the acid number (milligrams KOH per gram of oil).

(*f*) *Saponification Number.*—Weigh about 2 g. of the oil in a 300-ml. Erlenmeyer flask. Add 25 ml. of alcoholic sodium hydroxide solution. Put a condenser loop inside the neck of the flask and heat on the steam bath for 1 hr. Cool, add phenolphthalein as indicator, and titrate with 0.5 N H_2SO_4. Run two blanks with the alcoholic sodium hydroxide solution. These should check within 0.1 ml. of 0.5 N H_2SO_4. From the difference between the number of milliliters of 0.5 N H_2SO_4 required for the blank and for the determination, calculate the saponification number (milligrams KOH required for 1 g. of the oil).

(*g*) *Unsaponifiable Matter.*—Weigh 8 to 10 g. of the oil. Transfer to a 250-ml. long-neck flask. Add 5 ml. of a strong solution of NaOH (1:1) and 50 ml. of ethyl alcohol (95 per cent). Put a condenser loop inside the neck of the flask and boil for 2 hr. Occasionally agitate the flask to break up the liquid, but do not project the liquid onto the sides of the flask. At the end of 2 hr., remove the condenser and allow the liquid to boil down to about 25 ml.

Transfer to a 500-ml. glass-stoppered separatory funnel, rinsing with water. Dilute with water to 250 ml., and add 100 ml. of redistilled ether. Stopper and shake for 1 min. Let stand until the separation between the two layers is sharp and clear. Draw off all but one or two drops of the aqueous layer into a second 500-ml. separatory funnel and repeat the process, using 60 ml. of ether. After thorough separation, draw off the aqueous solution into a 400-ml. beaker, then transfer the ether solution to the first separatory funnel, rinsing down with a little water. Return the aqueous solution to the second separatory funnel and shake out again with 60 ml. of ether in a similar manner, finally drawing the aqueous solution into the beaker and rinsing the ether into the first separatory funnel.

Shake the combined ether solution with the combined water rinsings and let stand until the separation between the two layers is sharp and clear. Draw off the water and add it to the main aqueous solution. Shake the ether solution with two portions of water (about 25 ml. each). Add these to the main water solution.

Swirl the separatory funnel so as to bring the last drops of water down to the stopcock and draw off until the ether solution just fills the bore of the stopcock. Wipe out the stem of the separatory funnel with a bit of cotton on a wire. Draw off the ether solution (portionwise if necessary) into a 250-ml. flask and distill off. While still hot, drain the flask into a small weighed beaker, rinsing with a little ether. Evaporate this ether, cool the beaker, and weigh.

NOTE.—The unsaponifiable oil from adulterated drying oils may be volatile and as a consequence may evaporate on long heating. Therefore, heat the beaker on a warm plate, occasionally blowing out with a current of dry air. Discontinue heating as soon as the odor of ether is gone.

(*h*) *Iodine Number* (*Wijs*).—Place a small quantity of the sample in a small weighing burette or beaker. Weigh accurately. Transfer by dropping from 0.09 to 0.15 g. of oil to a 500-ml. bottle, having a well-ground glass stopper, or an Erlenmeyer flask, having a specially flanged neck for the iodine tests. Reweigh the burette or beaker and determine the amount of sample used. Add 10 ml. of chloroform. Whirl the bottle to dissolve the sample. Add 10 ml. of chloroform to each of two empty bottles like that used for the sample. Add to each bottle 25 ml. of the Wijs solution and let stand with occasional shaking for 1 hr. in a dark place at a temperature of from 21 to 23 C. Add 10 ml. of the potassium iodide solution (15 per cent) and 100 ml. of water. Titrate with 0.1 N sodium thiosulfate, using starch as an indicator. The titrations on the two blank tests should agree within 0.1 ml. From the difference between the average of the blank titrations and the titration on the samples and the iodine value of the thiosulfate solution calculate the iodine number of the samples tested. (Iodine number is given in centigrams of iodine to 1 g. of sample.)

(*i*) *Color.*—Prepare a fresh solution of pure potassium bichromate in pure colorless H_2SO_4 (sp. gr. 1.84). This solution should be in the proportion of 1.0 g. potassium bichromate to 100 ml. (184.0 g.) of H_2SO_4. Place the oil and colored solution in separate thin-walled, clear glass tubes of the same diameter (1 to 2 cm.) to a depth of not less than 2.5 cm. and compare the depths of color by looking transversely through the columns of liquid by transmitted light.

The iron-cobalt liquid equivalent to the above bichromate solution may be prepared as follows: Mix 29.4 ml. of Solution No. 1 with 17.6 ml. of Solution No. 2 and 53.0 ml. of Solution No. 3. The three base solutions referred to have the following composition:

Solution No. 1	$FeCl_3 . 6H_2O$	5.0 g.
	HCl (2 per cent)	1.2 g.
Solution No. 2	$CoCl_2 . 6H_2O$	1.0 g.
	HCl (2 per cent)	3.0 g.
Solution No. 3	Aqueous solution of HCl (2 per cent)	

The Lovibond equivalent to the above iron-cobalt solution shall be as follows: red 17.5, yellow 120.0, and blue 0.20.

The depth of liquid shall be 10.5 mm.

(*j*) *Appearance.*—If the oil is cloudy when cold, it shall become clear and transparent when heated at 65 C. for 5 min.

TENTATIVE METHOD OF TEST
FOR
RELATIVE DRY HIDING POWER OF PAINTS[1]

A.S.T.M. Designation: D 344 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1932; REVISED, 1937.

Scope

1. This method is intended for determining whether the relative dry hiding power of a given sample of paint is less than, equal to, or greater than that of a mutually agreed upon standard. An optional procedure is included which permits a quantitative determination of the relative dry hiding powers of two paints when they differ not too greatly in this respect.

Definition

2. Relative dry hiding power of a paint is the ability of that paint to reduce the contrast of a black and white surface to which it is applied and allowed to dry. It is quantitatively expressed in terms of the proportional spreading rate[2] of paint required to produce the same contrast reduction as obtained with the paint chosen as standard.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Balance.*—Regulation laboratory balance with capacity of 100 g. and sensitivity of 0.1 g.

(*b*) *Syringe.*—Syringe of 10-ml. capacity.

(*c*) *Paint Brush.*—A good grade paint brush, 1½ in. in width.

(*d*) *Container.*—A ½-pt. can or a 250-ml. beaker.

(*e*) *Test Surface.*—A smooth-surfaced paper chart having adjacent black and white areas and coated with a suitable varnish or lacquer so as to render the surface impervious and resistant to paint liquids. The black areas of the board shall have a diffuse reflection of less than 1 per cent. The white areas shall have a diffuse reflection of 75 per cent or greater. The white areas of the boards used for the standard paint and the sample shall not differ in brightness by more than 1 per cent. The chart shall have a test area of not less than 1 sq. ft. and any suitable regular geometrical design of the contrasting areas may be used.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] See the definition of "Spreading Rate" as given in the Standard Definitions of Terms Relating to Paint Specifications (A.S.T.M. Designation: D 16) of the American Society for Testing Materials, 1936 Book of A.S.T.M. Standards, Part II, p. 826.

(*f*) *Illumination.*—A reasonable intensity of diffused light, preferably northern skylight or a source approximating northern skylight.

Materials

4. A standard paint mutually agreed upon by the purchaser and the seller.

Procedure

5. (*a*) The weight per gallon of the standard and sample paints must be known and may be determined by any accepted method.

(*b*) Brushouts of the standard paint and the sample paint shall be prepared as described in Paragraph (*c*) at a convenient and suitable spreading rate mutually agreed upon by the purchaser and the seller and preferably lying within the range of 400 to 800 sq. ft. per gal.

(*c*) The standard paint shall be well stirred and the brush dipped into the paint and worked out on a suitable surface. The desired quantity of paint shall be taken up in the syringe, and the syringe and wet brush weighed in the empty container. The paint shall then be spread over the surface of the chart, the syringe returned to the empty container, and the paint brushed out uniformly over the test surface, care being taken to cover only the test area. The empty syringe, wet brush, and container shall again be weighed. The loss of weight represents the weight of paint applied to the test surface. A test chart shall then be prepared from the sample paint in the same manner and at the same spreading rate. Both painted test surfaces shall then be set aside to dry.

(*d*) After the test surfaces are thoroughly dry, they shall be placed side by side against a flat surface. The standard and sample brushouts shall be viewed in juxtaposition under a reasonable intensity of diffused light, preferably northern skylight or a source approximating northern skylight. The observer should view the brushouts from a distance of 5 to 10 ft.

Relative Dry Hiding Power

6. If the apparent contrast existing on the brushout of the sample paint is adjudged equal to or less than that existing on the brushout of the standard paint, the sample paint shall be said to meet the standard paint in relative dry hiding power as defined in Section 2.

Optional Procedure

7. (*a*) In many cases it is not sufficient to say that the sample paint is better or poorer than the standard, and the amount of difference must be determined quantitatively. The following optional procedure is recommended for determining quantitative differences in hiding power. It may be used in place of the procedure outlined in Section 5:

(*b*) The sample paint shall be applied to a brushout board as described in Section 5 (*c*) at the spreading rate mutually agreed upon or at a convenient spreading rate. A series of brushouts shall be made from the standard paint. The difference in spreading rate between successive steps in this series should be approximately 15 per cent. The range of spreading rate covered by this series should be sufficient to give contrasts above and below that of the brushout of the sample paint (estimated from the probable difference in relative dry hiding power, if known) or should cover the entire range of spreading rate convenient to apply with the standard paint. The painted test surfaces shall be set aside to dry.

(*c*) Under the conditions of illumination and viewing, as described in Section 5 (*d*), the brushout of the sample paint shall be compared with a series of brushouts of the standard paint, numbered, beginning with one, in order of decreasing spreading rate for convenience of handling. It will be found to be equal in contrast to one brushout of the standard or to be intermediate in contrast between two adjacent standards in the series of standard brushouts. If the sample is found to be between two adjacent standards in contrast, the sample shall be viewed against these standards with the higher and lower standard on either side. For purposes of grading, the contrast interval between the two standards shall be considered as 10 arbitrary units. The position of the sample shall be estimated on this arbitrary scale. Thus, if the sample appears equally distant in contrast from either standard, it will be graded 5 on the arbitrary scale. If nearer the standard of less contrast, it will be graded 6, 7, 8, or 9 as the case might be. The grading shall be recorded in terms of the number of the standard brushout of higher spreading rate followed by a decimal point and the grading of the arbitrary scale. In making this grading it is important that the sample be graded between the two standards with the standard of lesser contrast first on the right and then on the left. The two gradings thus obtained shall be averaged to give the final grading figure.

Hiding Power Calculation

8. The hiding power of the sample of paint shall be calculated as follows:

$$\text{Hiding power} = \frac{(W_1 - W_2)\frac{X}{10} + W_2}{W_3} \times \frac{S_2}{S_1} \times 100$$

where W_1 = the weight of paint on the brushout of the heavier grading standard,

W_2 = weight of paint on the brushout of the lighter grading standard,

W_3 = weight of paint on the brushout of the sample paint,

X = the contrast grading (decimal portion of the recorded grading),

S_1 = the weight per gallon of the standard paint, and

S_2 = the weight per gallon of the sample paint.

TENTATIVE METHOD OF TEST
FOR
SPECTRAL APPARENT REFLECTIVITY OF PAINTS[1]

A.S.T.M. Designation: D 307 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued as Tentative, 1929; Adopted in Amended Form, 1930;
Reissued as Tentative, 1937.

Scope

1. This method determines spectral apparent reflectivity of paints relative to magnesium oxide.

Definitions

2. (*a*) *Reflectivity.*—Reflectivity is the ratio of the reflected to the incident energy for a paint of sufficient thickness so that any increase would fail to change this ratio.

(*b*) *Apparent Reflectivity.*—Apparent reflectivity is the reflectivity which an ideal diffusing material would need to possess in order to yield the same appearance as the sample under the same illumination and viewing conditions.

Note.—The appearance of a painted surface is not completely defined by the terms in Paragraphs (*a*) and (*b*), but may also be influenced by other properties, such as texture and gloss.

Apparatus

3. The apparatus shall consist of a spectrophotometer by means of which the spectral distribution curve of the apparent reflectivity of a given paint sample relative to magnesium oxide may, within the limits of the visible spectrum, be determined. Such apparatus may be of either visual or photoelectric type, but shall be suitable for determinations throughout the visible spectrum. The apparatus shall be so designed as to provide for the elimination of stray energy, by such means as the introduction of stray energy filters or the use of two dispersing prisms.

Standard Reflecting Surface

4. The standard of apparent reflectivity shall be a layer (at least 0.5 mm. in thickness) of magnesium oxide, freshly prepared by collecting the smoke from burning magnesium ribbon or shavings on a suitable surface.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

This method is in effect a revision of and replaces the former Standard Method of Analysis for the Color Characteristics of Paints in Terms of Fundamental Physical Units (D 307 - 30), which standard was accordingly discontinued in 1937.

NOTE.—For further information regarding preparation of fumed standards, see National Bureau of Standards *Letter Circular 395*, "Preparation and Colorimetric Properties of a Magnesium Oxide Reflectance Standard," January 20, 1936.

Illumination

5. (*a*) The sample and standard shall be illuminated under identical conditions.

(*b*) The manner of illumination shall be specified, stating whether the illumination is diffuse, nearly diffuse, or nearly uni-directional. If nearly uni-directional, the direction and angular size of the source shall be specified.

(*c*) The direction and angular size of the viewing element shall be specified.

NOTE.—This method (measurement of spectral apparent reflectivity relative to a standard) serves to specify the optical properties of paint which are responsible for its color, subject, however, to the limitation that the color may depend upon the manner of illumination and viewing. For glossy samples in particular it is important to specify the manner of illumination and viewing, and the most essential single characteristic is whether or not the specular component of reflected energy is included in the measurement. Since paint samples are customarily examined for color in such a way as to avoid specularly reflected light as much as possible, it is recommended that in these measurements also, the specular component be excluded by proper choice of illuminating and viewing conditions (such as 45-deg. illumination with perpendicular viewing, as recommended in 1931 by the International Commission on Illumination) or by application of a suitable correction. It should be noted that the appearance of paint requires for its specification, in addition to the above, some measure of its gloss. Such a measure is beyond the intended scope of this method.

Calibrations

6. (*a*) The calibration of the photometer scale, or other intensity measuring unit, shall be carefully checked at reasonable time intervals in a manner to insure accuracy over the entire range.

(*b*) The accuracy of the wave length calibration shall be adequately checked at reasonable time intervals.

(*c*) The slit-width shall be adjusted so that slit-width corrections are negligible; otherwise these corrections shall be applied.

Procedure

7. (*a*) The sample shall be handled carefully to avoid discoloration, and care shall be taken not to touch the area to be tested. The condition of the sample before and after analysis shall be carefully noted and any change in the appearance of the surface of the tested area shall be recorded.

(*b*) Determination of spectral apparent reflectivity relative to the standard shall be made throughout the visible spectrum at sufficiently close intervals that the curve drawn by interpolation would not be changed by taking further readings.

NOTE.—Readings at 20 mμ. intervals between 440 mμ. and 700 mμ. are usually sufficient. Typical curves of flat and gloss paints in red, blue and gray are shown in Fig. 1.

(*c*) The paint on which spectral apparent reflectivity determinations are to be made, shall be applied to a given surface in a sufficient number of coats, so that any additional coats will not cause a change in appearance.

Record

8. The results obtained shall be preserved as a record.

Rejection

9. If the sample undergoes a visible change during the analysis, the readings shall be rejected (see Section 7 (*a*)).

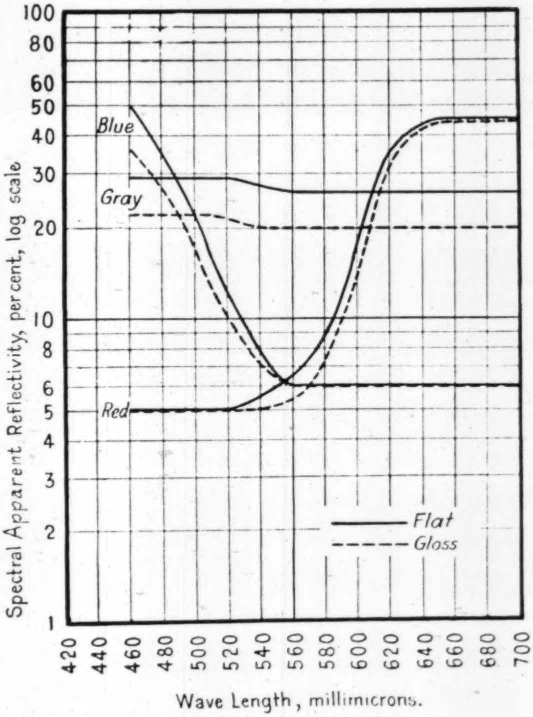

FIG. 1.—Typical Curves of Apparent Spectral Reflectivity.

Report

10. The report of the spectral apparent reflectivity of a paint sample shall consist of the following:

(*a*) The characteristic spectral apparent reflectivity of the sample relative to that of the standard, as specified in Sections 4 and 7.

(*b*) The manner of illumination and manner of viewing, as specified in Section 5 (*b*) and (*c*).

TENTATIVE SPECIFICATIONS
FOR
GASOLINE[1]

A.S.T.M. Designation: D 439 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.[2]

Scope

1. (*a*) These specifications are for the use of purchasing agencies in formulating specifications for purchases of gasoline under contract.

(*b*) These specifications are not a definition of gasoline, nor do they include all types of gasoline or motor fuel satisfactory for motor vehicles. Certain equipment or conditions of use may require fuels having special characteristics. Some trucks and buses, for example, may require lower vapor pressures and higher distillation temperatures than those included in these specifications.

Types

2. Three types of gasoline are provided, as follows:

Type A.—For use under normal conditions.

Type B.—For use where a gasoline of greater over-all volatility than Type *A* is desired.

Type C.—For use where a relatively non-volatile fuel is desired.

General

3. These specifications state the required properties of gasoline at the time and place of delivery in bulk. They provide for an automatic variation by the seller to meet the requirements of seasonal changes in temperature, depending upon the season and the locality in which the product is to be used. This is done by providing three vapor pressures and 10 per cent distillation points for each type specified and differentiating the use of these variations according to the months of the year and the geographical location by states.

Detailed Requirements

4. The three types of gasoline shall conform to the following requirements:

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Accepted for publication as tentative by Committee E-10 on Standards, March 30, 1937.

		TYPE A	TYPE B	TYPE C
Distillation Temperature, max., deg. Fahr., of:				
10 per cent evaporated	W[a]	140	140	167
	F[a]	149	149	
	S[a]	158	158	
50 per cent evaporated		284	257	284
90 per cent evaporated		392	356	392
Residue, max., per cent		2	2	2
Vapor pressure, max., lb. per sq. in. at 100 F	W[a]	13.5	13.5	13.5
	F[a]	11.5	11.5	11.5
	S[a]	9.5	9.5	9.5
Octane number, min		67 or 75[b]	67 or 75[b]	45[b]
Corrosion		passes	passes	passes
Gum, max., mg. per 100 ml		7[c]	7[c]	7[c]
Sulfur		[d]	[d]	[d]

[a] *W*, *F*, and *S* denote the automatic seasonal variations indicated in Table I.

[b] In all cases the octane number shall be agreed upon between the purchaser and the seller. In general, the following is applicable at the present time (1937):

75 octane number, minimum, corresponds to premium-priced gasolines.
67 octane number, minimum, corresponds to regular-priced gasolines.
45 octane number, minimum, corresponds to less than regular-priced gasolines.

[c] In the case of gasoline containing added non-volatile material, the gum requirement shall apply to the base stock.

[d] The technical data available do not afford an adequate basis for specifying maximum sulfur content. At the present time (1937), gasolines containing up to 0.25 per cent sulfur are distributed within the United States.

Basis for Purchase

5. The purchasing agency shall:

(*a*) State the minimum octane number as agreed upon with the seller (Section 4, footnote "b"),

(*b*) Specify the volatility, Type *A*, *B*, or *C*, desired, and

(*c*) Indicate the season and locality in which the fuel is to be used.

Methods of Testing

6. The requirements enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Distillation.*—Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine and Similar Petroleum Products (A.S.T.M. Designation: D 86).[1]

(*b*) *Vapor Pressure.*—Tentative Method of Test for Vapor Pressure of Petroleum Products (Reid Method) (A.S.T.M. Designation: D 323 – 37 T).[2]

(*c*) *Octane Number.*—Tentative Method of Test for Knock Characteristics of Motor Fuels (A.S.T.M. Designation: D 357 – 37 T).[3]

(*d*) *Corrosion.*—Standard Method of Test for Detection of Free Sulfur and Corrosive Sulfur Compounds in Gasoline (A.S.T.M. Designation: D 130).[4]

(*e*) *Gum.*—Standard Method of Test for Gum Content of Gasoline (A.S.T.M. Designation: D 381).[5]

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 870.

[2] See p. 888.

[3] See p. 879.

[4] 1936 Book of A.S.T.M. Standards, Part II, p. 973.

[5] *Ibid.*, p. 935.

TABLE I.—SCHEDULE FOR GEOGRAPHICAL SEASONAL VARIATIONS, W, F, AND S.

This schedule represents the time and place of use of the gasoline.
Shipments intended for future use may anticipate this schedule.
Where alternative variations are indicated, the option shall be exercised by the seller.

Territory	Month											
	January	February	March	April	May	June	July	August	September	October	November	December
Section 1	W	W	W or F	F	F or S	S	S	S	S or F	F	F or W	W
Section 2	W	W or F	W or F	F or S	F or S	S	S	S	S or F	F	F or W	F or W
Section 3	W	W or F	F or S	F or S	S	S	S	S	S	S or F	F	F or W
Section 4	F	F or S	F or S	S	S	S	S	S	S	S or F	S or F	F

SECTION 1

Idaho
Iowa
Maine
Michigan
Minnesota
Montana
New Hampshire
North Dakota
South Dakota
Vermont
Wisconsin
Wyoming

SECTION 2

Colorado
Connecticut
Delaware
District of Columbia
Illinois
Indiana
Kansas
Kentucky
Maryland
Massachusetts
Missouri
Nebraska
Nevada
New Jersey
New York
Ohio
Oregon
Pennsylvania
Rhode Island
Utah
Virginia
Washington
West Virginia

SECTION 3

Alabama
Arizona[a]
Arkansas
California
Georgia
Mississippi
New Mexico[a]
North Carolina
Oklahoma
South Carolina
Tennessee
Texas[a]

SECTION 4

Arizona[b]
Florida
Louisiana
New Mexico[b]
Texas[b]

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[a] North of 33 deg. latitude.
[b] South of 33 deg. latitude.

TENTATIVE METHOD OF TEST
FOR
DISTILLATION OF PLANT SPRAY OILS[1]

A.S.T.M. Designation: D 447 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. This method is intended for use in the distillation of plant spray oils.

Flask

2. The distillation flask shall be a Saybolt distilling flask, the dimensions and permissible variations being as follows:

	Centimeters	Inches	Tolerances, cm.
Diameter of bulb, outside	8.57	3.37	±0.3
Diameter of neck, inside	1.50	0.59	±0.1
Over-all length, neck plus bulb	21.50	8.46	±0.3
Length of vapor tube	17.50	6.89	±0.5
Diameter of vapor tube, outside	0.60	0.24	±0.05
Diameter of vapor tube, inside	0.40	0.16	±0.05
Thickness of vapor tube wall	0.10	0.04	±0.02

The center of the vapor tube shall be 6.5 cm. ± 0.3 cm. (2.56 in. ±0.12 in.) below the top of the neck. The tube is approximately in the middle of the neck and set at an angle of 75 deg., ±3 deg., with the vertical.

Condenser

3. The condenser[2] shall consist of a $\frac{9}{16}$-in. (14.29-mm.), outside diameter, No. 20 Stubbs Gage seamless brass tube, 22 in. (55.88 cm.) in length. It shall be set at an angle of 75 deg. with the vertical and shall be surrounded with a bath 15 in. (38.1 cm.) in length, approximately 4 in. (10.16 cm.) in width by 6 in. (15.24 cm.) in height. The bath shall be provided with suitable connections for circulating water through it. The lower end of the condenser tube shall be cut off at an acute angle, and curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the receiving graduate at a point approximately 1 to $1\frac{1}{4}$ in. (2.54 to 3.18 cm.) below the top of the graduate when it is in position to receive the distillate.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M Committee D-2 on Petroleum Products and Lubricants.

[2] A condenser conforming to these specifications is illustrated in the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86) of the American Society for Testing Materials, see 1936 Book of A.S.T.M. Standards, Part II, p. 870.

Shield

4. The shield[1] shall be made of approximately No. 22 gage sheet metal and shall be 19 in. (48.26 cm.) in height, 11 in. (27.94 cm.) in length and 8 in. (20.32 cm.) in width, with a door on one narrow side, with two openings, 1 in. (2.54 cm.) in diameter, equally spaced, in each of the two narrow sides, and with a slot cut in one side for the vapor tube. The centers of these four openings shall be $8\frac{1}{2}$ in. (21.59 cm.) below the top of the shield. There shall also be three $\frac{1}{2}$-in. (1.27-cm.) holes in each of the four sides with their centers 1 in. (2.54 cm.) above the base of the shield.

Support

5. The ring support may be of the ordinary laboratory type, 4 in. (10.16 cm.) or larger in diameter, and shall be supported on a stand inside the shield. There shall be two hard asbestos boards: One 6 by 6 by $\frac{1}{4}$ in. (15.24 by 15.24 cm. by 6.35 mm.) with a hole $2\frac{3}{4}$ in. (6.99 cm.) in diameter in its center, the sides of which shall be perpendicular to the surface; the other, an asbestos board to fit tightly inside the shield, with an opening 4 in. (10.16 cm.) in diameter concentric with the ring support. These shall be arranged as follows: The second asbestos board shall be placed on the ring and the first or smaller asbestos board on top so that it may be moved in accordance with the directions for placing the distilling flask. Direct heat shall be applied to the flask only through the $2\frac{3}{4}$-in. (6.99-cm.) opening in the first asbestos board. There shall also be provided a cover of $\frac{1}{4}$-in. hard asbestos board for the shield in two pieces so formed as to fit tightly around the neck of the distillation flask.

Heater

6. The burner shall be so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified below. The flame should never be so large that it spreads over a circle of diameter greater than $4\frac{1}{2}$ in. (11.43 cm.) on the under surface of the asbestos board. A sensitive regulating valve and gas pressure governor are desirable adjuncts, as they give complete control of heating.

Thermometer

7. The A.S.T.M. high-distillation thermometer, conforming to the requirements specified in Section 6 (*b*) of the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86) of the American Society for Testing Materials,[2] shall be used.

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[1] A shield conforming to these specifications is illustrated in the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86) of the American Society for Testing Materials, see 1936 Book of A.S.T.M. Standards, Part II, p. 870.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 870.

Graduate

8. The graduate shall be of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top. The cylinder shall be graduated to contain 100 ml. and the graduated portion shall be not less than 7 in. (17.78 cm.) nor more than 8 in. (20.32 cm.) in length. It shall be graduated in single milliliters and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom at intervals of 10 ml. The over-all height of the graduate shall be not less than 9¾ in. (24.8 cm.) nor more than 10¼ in. (26.0 cm.). The graduations shall not be in error by more than 1 ml. at any point on the scale.

Procedure

9. (*a*) The condenser tube shall be swabbed to remove any liquid remaining from the previous test. A piece of soft, lint-free cloth attached to a cord or copper wire may be used for this purpose.

(*b*) Exactly 100 ml. of the sample at a temperature of 70 to 80 F. (21 to 27 C.) shall be measured into the 100-ml. graduate. The sample shall then be poured into the distilling flask allowing the cylinder to drain 30 sec. The cylinder shall then be placed in a boiling-water bath for approximately 1 min., after which the remaining oil shall be drained into the distilling flask for 30 sec.

(*c*) The thermometer, provided with a cork, shall be fitted tightly into the neck of the flask so that the bulb will be in the middle of the neck and, so that the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask.

(*d*) The flask shall be placed over the 2¾-in. (6.99-cm.) opening in the asbestos board with the vapor outlet tube inserted into the condenser tube. A tight connection shall be made by means of a cork through which the vapor tube passes. The position of the flask shall be so adjusted that the vapor tube extends into the condenser tube not less than 1 in. (2.54 cm.) nor more than 2 in. (5.08 cm.). The neck of the distillation flask shall extend 2$\frac{5}{32}$ in. (5.50 cm.) above the cover of the shield.

(*e*) A clean, dry, graduated cylinder shall be placed at the outlet of the condenser tube in such a position that the condenser tube shall extend into the graduate approximately 1 in. (2.54 cm.) but not below the 100-ml. mark.

(*f*) When everything is in readiness, water shall be circulated through the condenser bath at such a rate that the water overflowing the condenser shall be at a temperature of approximately 140 F. (60 C.). The shield shall be covered with an asbestos-board cover during the distillation. Heat shall then be applied to the contents of the flask at a uniform rate, so regulated that the first drop of condensate falls from the condenser in not less than 10 nor more than 15 min. When the first drop falls from the end of the condenser tube, the reading of the distillation thermometer shall be recorded as the initial boiling point. The oil shall be allowed to drop directly into the cylinder and shall not flow down the side. The heat shall then be so regulated that the distillation proceeds at a uniform rate of not less than 4 nor more than 6 ml. per min. The vapor temperature shall be read every 5 ml. of condensate collected, the volumes being read at the temperature at which the oil is received in the graduate. The volume of condensate collected at any specified temperature shall also be read and recorded.

(*g*) The distillation shall be continued until 95 per cent of the sample has been distilled, unless cracking occurs. Cracking will be evidenced by an increase in the distilling rate with a thermometer reading that may advance very slowly, remain stationary, or recede, and an effort to adjust the distilling rate will usually result in a decided drop in the temperature reading. Should cracking occur, the distillation shall be immediately stopped, the temperature and volume of condensate being read and recorded.

Reproducibility of Results

10. With proper care and strict attention to details, duplicate determinations by the same operator should agree within ± 1 per cent of condensate on the basis of the original sample at specified temperatures above 600 F. (315 C.). Determinations by different operators should agree within ± 1.5 per cent.

Approved as
AMERICAN TENTATIVE STANDARD
by the American Standards Association
A.S.A. No.: Z11.37-1937

TENTATIVE METHOD OF TEST
FOR
KNOCK CHARACTERISTICS OF MOTOR FUELS[1]

A.S.T.M. Designation: D 357 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1933; REVISED, 1934, 1936, 1937.

Scope

1. This method is intended for determining the knock characteristics, in terms of an arbitrary scale of octane numbers, of gasolines and equivalent fuels for use in spark-ignition engines, other than engines for aircraft.

A.S.T.M. Octane Number

2. The A.S.T.M. octane number of a motor fuel is the whole number nearest to the octane number (Note 1) of that mixture of *iso*octane with normal heptane which the motor fuel matches in knock characteristics when compared by the procedure specified herein.

NOTE 1.—Octane number is defined by and is numerically equal to the percentage by volume of *iso*octane (2,2,4-trimethylpentane) in a mixture of *iso*octane and normal heptane, used as a primary standard for measurement of knock characteristics. Thus, by definition, normal heptane has an octane number of zero and *iso*octane of 100.

APPARATUS

Apparatus

3. The knock-testing unit described in this section shall be used without modification. The engine shall be known as the "C.F.R. Engine" and shall be marked[2] by plate or other approved means with a combination of the respective emblems of the American

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[1] This method heretofore known as the C.F.R. Motor Method is based on the apparatus and procedure developed by the Cooperative Fuel Research Committee, composed of representatives of the American Petroleum Institute, National Automobile Chamber of Commerce, Society of Automotive Engineers and U. S. Bureau of Standards, who have been studying this problem since 1928.

Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] At present the sole authorized manufacturer of the C.F.R. Engine is the Waukesha Motor Co., Waukesha, Wis. Other manufacturers may be approved in the future, but testing laboratories should not purchase engines except from Waukesha Motor Co. without ascertaining whether such engines have been approved. Inquiries in this connection should be directed to Mr. R. P. Anderson, secretary of Committee D-2 on Petroleum Products and Lubricants, 50 West 50th St., New York City.

Society for Testing Materials and the Cooperative Fuel Research Committee, thus:

ASTM=CFR

The apparatus shall consist of a continuously-variable-compression motor together with suitable loading and accessory equipment as follows:

(*a*) *Engine.*—Continuously-variable-compression, one-cylinder, with dimensions as follows:

Bore, in	3.25
Stroke, in	4.50
Displacement, cu. in	37.4
Valve diameter, in. clear	1.1875
Connecting-rod bearing:	
diameter, in	2.25
length, in	1.625
Front main bearing:	
diameter, in	2.25
length, in	2.00
Rear main bearing:	
diameter, in	2.25
length, in	4.25
Piston pin, floating, diameter, in	1.25
Connecting rod, center to center, in	10.00
Timing gear face, in	1.00
Piston rings, number	5
Exhaust pipe, diameter, in	1.25
Spark plug, size, mm	18.00
Weight of engine, lb. (approximate)	475
Weight of complete unit, lb. (approximate)	1375

(*b*) *Crankshaft.*—Fully machined, heat-treated, and counterbalanced.

(*c*) *Crankcase.*—Cast iron, with rigid end walls.

(*d*) *Connecting Rod.*—Rifle-drilled, S.A.E. No. 1045 steel, heat-treated, bearing alloy cast directly into big end.

(*e*) *Main Bearings.*—Renewable sleeve bushings, babbitt-lined.

(*f*) *Valves.*—Silcrome. Inlet valve with specially designed shroud.

(*g*) *Push Rods.*—Mushroom type with lock-nut adjustment.

(*h*) *Cylinders.*—Cast of iron alloy, bored and honed, Brinell hardness, 220 ± 20.

(*i*) *Cylinder Head.*—Integral with cylinder.

(*j*) *Cooling System.*—Evaporatively cooled.

(*k*) *Lubrication.*—Pressure feed to main connecting-rod, piston-pin, and camshaft bearings, and to idler gear stud and gears.

(*l*) *Oil Heater and Thermometer.*—Electric heater in base to bring oil to operating temperature quickly, and a thermometer on the instrument panel to indicate when equilibrium temperature has been reached.

(*m*) *Ignition.*—May be either a battery system or a magneto. A neon-tube spark indicator is built into the engine. Spark advance is automatically adjusted as the compression ratio is changed.

(*n*) *Carburetor.*[1]—A special C.F.R. multiple-bowl carburetor is furnished. The carburetor has a fixed fuel jet, and each float bowl is individually adjustable for level with respect to the jet for varying mixture ratio. Fuel containers are furnished with the carburetor.

(*o*) *Mixture Heater.*—As supplied by manufacturer, consisting of (*1*) a manifold, (*2*) an electric immersion heater and rheostat for controlling mixture temperature and (*3*) a special mercury stem thermometer of suitable range, so mounted that the bulb is centered in the mixture stream to indicate the mixture temperature.

(*p*) *Instruments.*—Knock intensity is measured by a bouncing pin, in conjunction with a knockmeter. (The knockmeter is a damped hot-wire ammeter which indicates the effective current in the circuit, thus permitting instantaneous readings.) Current is supplied from a small direct-current generator, belt-driven from the power-absorbing unit.

(*q*) *Power-Absorbing Unit.*—The engine is connected by V-type belts to an electric generator. This preferably should be an induction motor with synchronous characteristics but may be any electric generator capable of maintaining proper operating conditions. In most cases the electric generator will act as a starting motor to crank the engine, but if a direct-current generator is used and no outside source of current is available, the engine may have to be cranked by hand.

(*r*) *Complete Unit.*—The complete unit may be obtained with the engine, generator, and panel board mounted on a cast-iron base plate. All necessary instruments and accessories are furnished with the unit.

Foundation

4. The foundation shall be of concrete and at least 18 in. high. If possible, it should be prepared by pouring on a concrete floor. Proper dimensions will be furnished with the engine. For convenience in operation, the foundation should be placed at least 2 ft. from any

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[1] The use of knock-testing apparatus equipped with the carburetor described in the 1933 edition of the method (*Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 746 (1933); also 1933 Book of A.S.T.M. Tentative Standards, p. 540) is permissible.

wall. Should the floor of the building on which the engine is to be installed be other than portland-cement concrete, the floor should be cut away so that the engine foundation will rest directly on the ground. In such case the bearing area of the foundation will depend upon the condition of the underlying soil and should be such as to give ample footing.

Reference Fuels

5. (*a*) *Primary Reference Fuels.*—The primary[1] reference fuels shall be *iso*octane (2,2,4-trimethylpentane), and normal heptane. Both shall be certified for suitability as primary reference fuels by the National Bureau of Standards (Note 1).

(*b*) *Secondary Reference Fuels.*—To insure a greater degree of reproducibility in testing, it is preferable to use secondary[2] reference fuels which have been properly calibrated against the primary reference fuels on a large number of engines. Such secondary reference fuels may be straight-run or other stable gasolines suitable for the purpose. One of the reference fuels should be of low knock rating and the other of high knock rating. These secondary reference fuels shall be calibrated against normal heptane and *iso*octane. The calibration shall be expressed in terms of the octane-number scale, shall be based on results by this method from at least ten laboratories, and shall be certified by the National Bureau of Standards (Note 2).

NOTE 1.—For the present this certification consists of tests made by the Bureau of Standards on each batch of normal heptane or *iso*octane prepared respectively by the two companies mentioned,[1] with certificates issued to these companies authorizing them to guarantee to the purchaser that the material shipped is a part of a batch so tested and to quote the results of the Bureau of Standards' tests.

NOTE 2.—For the present this certification is based on tests made by members of the C.F.R. Motor Fuels Committee on samples from each batch of secondary reference fuels, and on blends of the high and low secondary fuels against blends of *iso*octane and normal heptane. A certificate is then issued to the producers authorizing them to guarantee to the purchasers that the material shipped is a part of a batch so tested, and to quote the results of the tests.

Standard Operating Conditions

6. The engine shall be run under the following standard conditions:

(*a*) *Engine Speed.*—900 r.p.m. ± 3 r.p.m.

(*b*) *Jacket Temperature.*—Constant within ± 1° F. (0.6° C.) and at a temperature between the limits of 205 and 215° F. (96 and 102° C.).

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[1] **At present, *iso*octane may be obtained from Rohm & Haas Co., 222 W. Washington Square, Philadelphia, Pa., and normal heptane from the California Chemical Co., 220 Bush Street, San Francisco, Calif.**

[2] Such secondary reference fuels are commercially available through the Standard Oil Company of New Jersey with calibration curves as determined in accordance with this method, giving octane numbers for these fuels blended with each other. They may be ordered from the Standard Oil Development Co., Elizabeth, N. J.

(*c*) *Cooling Liquid.*—Distilled water, rain water, or ethylene-glycol solution when necessary at high altitude.

(*d*) *Crankcase Lubricating Oil.*—S. A. E. 30.

NOTE.—The viscosity range of crankcase lubricating oil, S.A.E. 30, is from 185 to 255 seconds when determined on the Saybolt Universal viscosimeter at 130° F. (54.4° C.), in accordance with the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88) of the American Society for Testing Materials.[1]

(*e*) *Oil Pressure.*—25 to 30 lb. per sq. in. under operating conditions.

(*f*) *Oil Temperature.*—The electric oil heater shall be used only to raise the oil temperature rapidly to the equilibrium operating temperature, normal operation maintaining the equilibrium thereafter. By "equilibrium" is meant that steady temperature attained by the oil upon continuous operation of the engine without the use of the heater.

(*g*) *Valve Clearances.*—Intake 0.008 in., cold; exhaust 0.010 in., cold.

(*h*) *Spark Advance.*—Automatically controlled:

26.0 deg. at 5:1 compression ratio (basic setting);
22.0 deg. at 6:1 compression ratio;
19.0 deg. at 7:1 compression ratio.

(*i*) *Breaker-Point Clearance.*—Battery system 0.015 in.; magneto 0.020 in.

(*j*) *Spark Plug.*—Shall conform to the standard metric plug having the tolerances and thermal characteristics equal to the No. 8 spark plug furnished by the Champion Spark Plug Co., Toledo, Ohio. Gap setting, 0.025 in.

(*k*) *Throttle Opening.*—All tests shall be conducted with the throttle opening at the point of maximum volumetric efficiency, approximately 90 on the throttle scale.

(*l*) *Carburetor Adjustment.*—For maximum knock.

(*m*) *Exhaust Pipe.*—A separate exhaust pipe should be used for each engine. This pipe should be made from 1¼-in. pipe having a maximum of two ells with a total length not to exceed 20 ft. The use of a short straight-through muffler of 1¼-in. diameter passage for prevention of noise is permissible.

(*n*) *Mixture Temperature.*—The mixture temperature shall be maintained at 300° F. ±2° F. (149° C. ±1.1° C.) as indicated by the mercury stem thermometer.

(*o*) *Bouncing Pin Assembly.*—The gap setting shall be 0.003 in. to 0.005 in.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 982.

NOTE.—The following instructions for setting of the bouncing-pin contacts should be used: With the daily inspection, observe the contact points and electrical connections to see that the points are smooth and that all the connections are tight. The gap setting should be checked (0.003-in. to 0.005-in.). The flat spring of the lower contact should touch the insulated pin with slight pressure. Too much pressure will reduce its sensitivity. To adjust the pressure accurately, set the points with 0.003-in. to 0.005-in. clearance. Then remove the diaphragm and bouncing pin. Bend the lower spring until there is from $\frac{3}{64}$-in. to $\frac{1}{16}$-in. gap between the points. Remove the upper stop-adjusting screw and bend the upper spring until there is $\frac{5}{32}$-in. gap between the points. Check the tension on the small plunger spring in upper stop-adjusting screw and see that it has from 1- to $1\frac{1}{4}$-lb. initial tension. This can be measured by pressing it against any convenient platform scale. The pin should then be re-assembled and the adjusting screw set to give 0.003-in. to 0.005-in. gap between the points. The final adjustment is made by setting the clearance so that a knockmeter reading between 50 and 60 is obtained when operating the engine at the proper knock intensity.

PROCEDURE

Starting and Stopping the Engine

7. While the engine is being turned over by electric motor the ignition shall be turned on and the carburetor set so as to draw fuel from one float bowl. To stop the engine the fuel and the ignition switch shall be turned off and then the motor shall be stopped by means of the push-button switch. To avoid corrosion of valves and seats between operating periods the engine should be turned over by hand until both valves are closed.

Preliminary Adjustment of Compression Ratio

8. Using a mixture of 65 parts of *iso*octane and 35 parts of normal heptane, the compression ratio for first audible knock shall be obtained by increasing the compression ratio, by increments of two turns of the crank, from a point where there is no knock to the compression ratio at which audible knock is first detected. The proper knock intensity for use in making knock ratings shall be the knock intensity obtained with the mixture of 65 parts of *iso*octane and 35 parts of normal heptane when the compression ratio is increased one unit over that compression ratio giving first audible knock. Then the numerical indication of knock intensity obtained from the knockmeter shall be recorded. This procedure is necessary for the first adjustment only.

NOTE.—This knock intensity should be equivalent to the intensity obtained with a mixture of 65 per cent of *iso*octane and 35 per cent of normal heptane at a compression ratio of 5.3 ± 0.05 to 1 when testing at a barometric pressure of 760 mm.

For subsequent tests on fuel samples the compression ratio shall be set to duplicate the knock intensity as recorded above, provided no change has been made in the bouncing-pin adjustment in the meantime. In no case shall the knock intensity be such that the engine does not cease firing when ignition is interrupted.

Outline of Procedure

9. The octane number of a fuel shall be ascertained by comparing the knock intensity for the fuel with those for various blends of the reference fuels until two blends differing in knock rating by not more than two octane numbers are found, one of which gives a higher knock intensity than the fuel and the other a lower knock intensity. The knock intensity shall be measured by a bouncing-pin indicator in conjunction with a knockmeter.

Before the test sample and the blends of the reference fuels can be compared, the compression ratio must be set to give the proper knock intensity and the carburetor adjusted to give the maximum knock for each fuel.

Adjustment of Carburetor for Test Fuel

10. Using the fuel whose knock rating is to be determined, the carburetor shall be adjusted as follows: After one float bowl of the carburetor has been filled with the fuel of which the octane number is to be determined, adjustment shall be made to obtain maximum knock by raising and lowering the level of the float bowl and noting whether the knockmeter reading increases or decreases. The float bowl shall then be moved in the direction in which the knock increases until the knock passes through a maximum. This point shall be checked three times and the float bowl set at the position of maximum knock. The knockmeter needle shall be allowed to reach equilibrium after each adjustment of the float-bowl level.

Final Adjustment of Compression Ratio

11. Finally, adjust the compression ratio to give the same reading on the knockmeter when using the fuel under test as was obtained in the first adjustment under Section 8.

12. A trial blend[1] of the low-octane-number reference fuel and the high-octane-number reference fuel, based on the expected knock rating of the fuel sample under test, shall be placed in another carburetor float bowl and the engine run on this trial blend. The level of this float bowl shall then be adjusted to the maximum knock position in a manner similar to that described in Section 10.

Octane Number Determination

13. With the carburetor set for the air-fuel ratio of maximum knock, alternate series of readings of knock intensity shall be taken on the fuel under test and on a reference fuel blend. The knockmeter

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[1] Extreme care is required to insure thorough mixing of ingredients.

needle shall be allowed to reach equilibrium before the final reading is recorded.

At least 3 alternate series of readings shall be taken on each fuel. After changing from one fuel to another, at least 1 minute shall be allowed for the engine to reach equilibrium. With some fuels an appreciably longer time interval may be required. If the average knock intensity of the fuel sample is higher than the average of the reference fuel blend, the test shall be repeated with a blend containing a decreased proportion of the high-octane-number reference fuel.[1] The test shall be continued in this manner until the knock intensity for the fuel sample is definitely higher than one blend and lower than another blend of the reference fuels. The difference between these two final reference fuel blends shall be not more than two octane numbers.

CALCULATION

Calculation

14. The knock rating of the fuel sample shall be obtained by interpolation from the figures so recorded and the nearest whole number shall be reported as A.S.T.M. octane number.

Check of Test Conditions

15. Test conditions shall not be regarded as standard unless a blend by volume of 65 per cent *iso*octane and 35 per cent normal heptane is matched under the specified procedure by a blend by volume of 68 ± 1 per cent one-degree benzene with 32 ± 1 per cent normal heptane. At atmospheric pressure of 760 mm. a compression ratio of approximately 5.3:1 is correct for this determination. Substantial variations in atmospheric pressure will change the compression ratio required.

NOTE 1.—For routine checking purposes, one-degree benzene and secondary reference fuels may be used.

NOTE 2.—"One-degree" benzene[2] is a commercial product conforming to the following requirements:

(*a*) Boiling range not greater than 1° C., embracing the boiling point of chemically pure benzene.

(*b*) Specific gravity of from 0.882 to 0.886, when determined in accordance with the Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (A.S.T.M. Designation: D 287) of the American Society for Testing Materials.[3]

(*c*) Free from hydrogen sulfide, carbon disulfide and thiophene.

(*d*) Free from turbidity, with color not darker than a solution of 3 mg. of potassium dichromate in 1 liter of water when compared in 50-ml. Nessler tubes.

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[1] When changing fuels in the containers, the fuel in the container shall be drained completely by means of the drain-cock provided. A small portion of the new fuel shall be allowed to flow through the line before closing the drain-cock.

[2] "One-degree" benzene may be procured from The Barrett Co., 40 Rector St., New York City.

[3] 1937 Supplement to Book of A.S.T.M. Standards, p. 121.

ACCURACY

Accuracy

16. Results obtained by this procedure with different C. F. R. engines and in different laboratories should differ by not more than two octane numbers.

While the accuracy of this method is as above stated, its sensitivity may permit detection of differences as small as 0.2 octane number when alternate readings of knock intensity are taken on the two fuels as directed in Section 13.

MAINTENANCE

Maintenance

17. It is recommended that a systematic inspection of the testing apparatus be carried out with the utmost care after each 50 hours of operation. The following points should be emphasized:

(*a*) *Bouncing Pin.*—Lack of sensitivity, fluctuating knockmeter reading, or irregular gas evolution may indicate diaphragm trouble. The diaphragm retaining nut should be tightened and any carbon accumulation removed. If this does not correct the condition, the diaphragm may have deteriorated and may need to be replaced.

(*b*) *Breaker Points.*—If necessary, pits should be removed and breaker-point clearance adjusted.

(*c*) *Spark Plugs.*—The porcelain should be carefully inspected for cracks, and defective plugs replaced. The spark gap shall be carefully checked and adjusted if necessary. When plugs are re-used any carbon deposit on the metal body of the plug or on the electrodes shall be removed mechanically before making gap adjustments.

(*d*) *Fuel System.*—Any foreign matter in the fuel containers, float bowls, lines, or carburetor shall be removed. Blowing with air and then flushing the fuel system with gasoline or benzol is a convenient method.

(*e*) *Cooling System.*—Any leaks revealed by inspection shall be repaired.

(*f*) *Combustion Chamber.*—Carbon deposits shall be removed by scraping.

(*g*) *Compression Pressure.*—The cause of any abnormal change between inspections shall be investigated and corrected.

(*h*) *Valves.*—Valves shall be removed and reground unless there is a continuous polished line of contact on both the valve and the valve seat. After reassembling, the valve-tappet clearances shall be carefully adjusted.

(*i*) *Piston Rings.*—Carbon shall be removed from rings and grooves.

(*j*) The intake manifold and heater blades shall be inspected and cleaned if necessary.

TENTATIVE METHOD OF TEST

FOR

VAPOR PRESSURE OF PETROLEUM PRODUCTS (REID METHOD)[1]

A.S.T.M. Designation: D 323 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1930; Revised, 1931, 1932, 1935, 1937.

Scope

1. This method is intended for the determination of the vapor pressure of volatile, non-viscous petroleum products.

Note.—Two sampling procedures are described in this method, one for products having vapor pressures of 26 lb. or less and the other for products of higher vapor pressures. Sections 10 to 15, inclusive, shall be followed for products in the first group, and Sections 10, 21 and 22 for products in the second group. In case of doubt, the maximum vapor-pressure specification of the product to be tested shall determine which sampling procedure to employ without regard to the numerical value obtained in the test.

Apparatus

Vapor-Pressure Bomb

2. The vapor-pressure bomb consisting of two sections or chambers, an upper section, or air chamber, and a lower section, or gasoline chamber, shall conform to the following requirements:

(*a*) *Air Chamber*.—The upper section, or air chamber, shown in Fig. 1, shall be a cylindrical vessel 2 to $2\frac{1}{8}$ in. in diameter and 10 in. $\pm\frac{1}{8}$ in. in length, inside dimensions. In one end of the air chamber a $\frac{1}{4}$-in. standard pipe thread shall be tapped, to receive the gage coupling. Other means of connecting the air chamber with the pressure gage may be employed, provided the requirements of Paragraphs (*e*) and (*f*) are fulfilled. In the other end of the air chamber an opening approximately $\frac{1}{2}$ in. in diameter shall be provided for purposes of coupling with the gasoline chamber. The nature of this opening will depend upon the method of coupling employed. The air chamber must drain completely from either end when held in a vertical position and for this reason the ends may be slightly convex.

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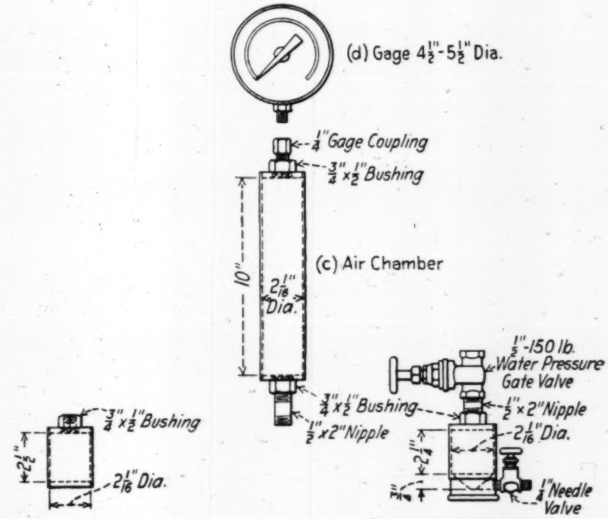
[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

This method comprises a consolidation of, and replaces the former Tentative Method of Test for Vapor Pressure of Natural Gasoline (Reid Method) (A.S.T.M. Designation: D 323 – 32 T) and the Tentative Method of Test for Vapor Pressure of Motor and Aviation Gasoline (Reid Method) (A.S.T.M. Designation: D 417 – 35 T), which methods were accordingly discontinued in 1937.

(*b*) *Gasoline Chamber.*—The lower section, or gasoline chamber, shown in Fig. 1 (*a*), shall be a cylindrical vessel of the same inside diameter as the air chamber and 2½ in. ± $\frac{1}{16}$ in. in inside length. In one end of the gasoline chamber an opening approximately ½ in. in diameter shall be provided for purposes of coupling with the air chamber. The nature of this opening will depend upon the method of coupling employed. The other end of the gasoline chamber shall be completely closed. The gasoline chamber must drain completely when inverted and for this reason the top may be slightly convex.


(d) Gage 4½"-5½" Dia.
¼" Gage Coupling
¾" x ½" Bushing
(c) Air Chamber
10"
2$\frac{1}{16}$" Dia.
¾" x ½" Bushing
½" x 2" Nipple
½"-150 lb. Water Pressure Gate Valve
½" x 2" Nipple
2$\frac{1}{16}$" Dia.
2¼"
¼" Needle Valve
¾" x ½" Bushing
2½"
2$\frac{1}{16}$" Dia.

(a) Gasoline Chamber with One Opening

(b) Gasoline Chamber with Two Openings

FIG. 1.—Vapor-Pressure Bomb, Screw-Coupling Type.

(*c*) *Gasoline Chamber for Sampling from Closed Vessels.*—For this use the lower section, or gasoline chamber, shown in Fig. 1 (*b*), shall be the same as described in Paragraph (*b*) except that a ¼-in. needle valve shall be attached near the bottom of the gasoline chamber and a ½-in. gate valve shall be introduced in the coupling between the chambers as illustrated in Fig. 1 (*b*). The vertical dimensions of the gasoline chamber proper in Fig. 1 (*b*) shall be such that the assembly fulfills the requirements of Paragraph (*e*) for the type of construction illustrated. The type of gasoline chamber shown in Fig. 1 (*b*) may be used for immersion sampling.

(*d*) *Method of Coupling Air and Gasoline Chambers.*—Any method of coupling the air and gasoline chambers may be employed, for example, a simple thread, a union, or a clamp. The sole provision in this respect is that the assembly shall be free from leaks under the conditions of test. Responsibility for freedom from leaks rests with the user.

(*e*) *Volumetric Capacity of Air and Gasoline Chambers.*—The ratio of the volume of the air chamber to the volume of the gasoline chamber shall be between the limits of 3.8 and 4.2. To determine the volume of the air chamber, insert a $\frac{1}{4}$-in. plug in the gage coupling, invert the chamber, fill with water up to and including the portion of the coupling device attached permanently to the air chamber and then carefully measure the volume of water. To determine the volume of the gasoline chamber, fill with water up to and including the portion of the coupling device attached permanently to the gasoline chamber, and then carefully measure the volume of water.

(*f*) *Checking for Freedom from Leaks.*—Before placing new apparatus in service and as often as necessary thereafter, the assembled vapor-pressure bomb shall be checked for freedom from leaks by filling with air to 100-lb. gage pressure and completely immersing in a water bath. Only apparatus which will stand this test without leaking shall be used.

Pressure Gage

3. (*a*) The pressure gage shall be a Bourdon-type spring gage of test-gage quality $4\frac{1}{2}$ to $5\frac{1}{2}$ in. in diameter. The range of the pressure gage used shall be governed by the vapor pressure of the sample being tested, as follows:

VAPOR PRESSURE, LB. PER SQ. IN. AT 100 F.	GAGE TO BE USED[a]		
	SCALE RANGE	NUMBERED INTERVALS	INTERMEDIATE GRADUATIONS
12 and under	0 to 15 lb.	3 lb.	0.1 lb.
10 to 36 lb	0 to 45 lb.	5 lb.	0.2 lb.
30 to 55 lb	0 to 60 lb.	10 lb.	0.25 lb.
50 and higher	0 to 100 lb.	10 lb.	0.5 lb.

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[a] A gage of 0 to 30-lb. range, 5-lb. numbered intervals and 0.2-lb. intermediate graduations may be used for products of from 10 to 26-lb. vapor pressure.

(*b*) Only accurate gages shall be continued in use.

Cooling Bath

4. A cooling bath shall be provided of such dimensions that the sample containers and gasoline chambers may be completely immersed. Means for maintaining the bath at a temperature of 32 to 40 F. shall be provided.

Water Bath

5. The water bath shall be of such dimensions that the vapor-pressure bomb may be immersed to at least 1 in. above the top of the air chamber. Means for maintaining the bath at a constant temperature of 100 F. ±0.2 F. shall be provided.

Water-Bath Thermometer

6. The thermometer for measuring the water-bath temperature shall conform to the following requirements:

TYPE: Etched stem, glass.
LIQUID: Mercury.
RANGE AND SUBDIVISIONS: Range 94 to 108 F., subdivisions in 0.2 F.
TOTAL LENGTH: 252 to 256 mm. (9.92 to 10.08 in.).
STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 mm. (0.24 to 0.28 in.).
BULB: Corning normal or equally suitable thermometric glass.
Length, 25 to 35 mm. (1.0 to 1.4 in.).
Diameter, not less than 5 mm. (0.20 in.) and not greater than that of stem.
DISTANCE TO LOWEST SPECIFIED GRADUATION FROM BOTTOM OF BULB: 135 to 150 mm. (5.3 to 5.9 in.).
DISTANCE TO HIGHEST SPECIFIED GRADUATION FROM TOP OF THERMOMETER: 20 to 35 mm. (0.8 to 1.4 in.).
CONTRACTION CHAMBER: To be of long narrow type, top to be not more than 60 mm. (2.36 in.) above bottom of bulb, mercury to stand in contraction chamber at 32 F.
EXPANSION CHAMBER: To permit heating the thermometer 90 F. above highest temperature on scale, and in all cases to permit heating to 212 F.
FILLING ABOVE MERCURY: Nitrogen gas.
TOP FINISH: Glass ring.
GRADUATION: All lines, figures and letters clear cut and distinct. The first and each succeeding 1 F. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 2 F.
IMMERSION: Total.
SPECIAL MARKING: The A.S.T.M. Reid-vapor-pressure test point of 100 F. to be numbered in red, other graduations and figures in black. A serial number and the manufacturer's name or trade mark shall be etched on the stem.
SCALE ERROR: The error at any point of the scale shall not exceed 0.2 F.
STANDARDIZATION: The thermometers are to be standardized for the condition of total immersion. Correction for emergent stem shall not be applied.
CASE: Each thermometer shall be supplied in a suitable case on which shall appear the marking "A.S.T.M. Reid-Vapor-Pressure Thermometer."

Air Chamber Thermometer

7. The thermometer for measuring the temperature of the air chamber shall conform to the following requirements:

TYPE: Etched stem, glass.
LIQUID: Mercury.
TOTAL LENGTH: 254 to 305 mm. (10.0 to 12.0 in.).
RANGE AND SUBDIVISIONS: Range −40 F. or −30 F. to + 120 F. or + 130 F.; subdivisions in 1 F.
STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6 to 7 mm. (0.24 to 0.28 in.).
BULB: Corning normal or equally suitable thermometric glass.
Length, not over 25 mm. (1.0 in.).
IMMERSION: Total.
SCALE ERROR: The error at any point of the scale shall not exceed 0.5 per cent of the scale range.

Chilled Sampling Apparatus

8. The chilled sampling apparatus shall consist of a bath of sufficient size to accommodate both the sample container and a cooling coil formed of $\frac{1}{4}$-in. outside diameter, No. 20 gage copper tubing. One end of the cooling coil shall be provided with connections suitable for attaching to the tank sampling connection and the other end shall be provided with a $\frac{1}{8}$-in. needle valve of good quality. For introducing the sample into the container this valve shall have at its open end a removable tube of $\frac{1}{4}$-in. outside diameter copper tubing of sufficient length to reach the bottom of the sample container. A bath of suitable design is shown in Fig. 2.

Sample Containers and Transfer Connections

9. The two types of containers for obtaining samples of gasoline for vapor-pressure tests shall conform to the following general requirements: The containers shall be at least one quart in size and of ample strength to withstand the pressures to which they may be subjected. The construction shall be such that the containers may be easily and securely sealed. Container openings in all cases shall be of a type which will permit the replacement of the cap, stopper, or plug with the connections provided for transferring samples to the gasoline chamber.

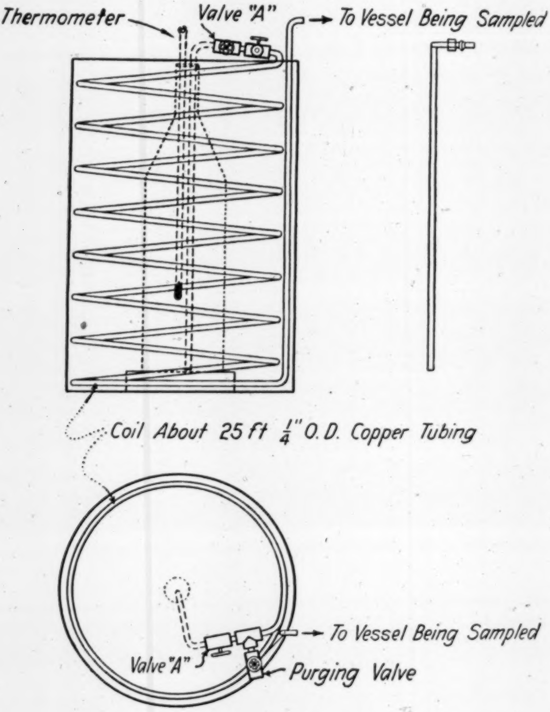


FIG. 2.—Chilled Sampling Apparatus.

(*a*) *Open-Type Containers.*—Containers classed as open-type containers shall be provided with a single opening. Such containers shall be of a type which will readily permit sampling by immersion.

The sample-transfer connection for the open-type container shall consist of two tubes assembled in a replacement cap or stopper. One of these tubes, the air tube, shall extend to the bottom of the sample container. The second tube, the liquid-delivery tube, shall be flush with the inside face of the cap or stopper and shall extend beyond the cap or stopper for a sufficient length to reach to the bottom of the gasoline chamber in the sample-transfer operation.

(*b*) *Closed-Type Containers.*—Containers classed as closed-type containers shall be provided with two openings, one in each end or the equivalent thereof, fitted with suitable valves.

The sample-transfer connection for the closed-type container shall consist of a single tube with fittings suitable for connection to one of the openings of the sample container, the tube being of sufficient length to reach to the bottom of the gasoline chamber in the sample-transfer operation.

SAMPLING

10. The following general remarks apply to all sampling for Reid-vapor-pressure determinations. The extreme sensitivity of vapor-pressure measurements to losses through evaporation and to slight changes in composition is such as to require the utmost precautions and the most meticulous care both in the obtaining and in the handling of samples to insure their being representative. Official samples shall be taken by, or under the immediate direction of, a person of judgment, skill and previous experience in sampling for vapor-pressure measurements.

Sampling Open Tank Cars and Small Open Tanks

11. Open-type sample containers (Section 9) shall be used for sampling open tank cars and small open tanks. The empty sample container shall first be immersed in the car or tank of liquid to be tested and allowed to fill while being lowered and raised through the liquid. The first filling is for the purpose of washing the container thoroughly. The container shall be emptied and then refilled. On this second filling the sample container shall be lowered and raised at such a rate that it will be nearly but not quite full when withdrawn. Enough of the contents shall be poured off immediately so that the portion remaining shall fill the sample container to not less than 70 per cent and not more than 80 per cent of its capacity. The sample container shall be promptly sealed.

Sampling Other Vessels

12. For obtaining samples from other sizes or types of vessels than those described in Section 11, the following general remarks apply: The methods of sampling, points of sampling, and number of samples shall be as agreed upon by the contracting parties but in all cases the procedure followed shall result in obtaining representative samples. Because of the sensitivity of this method of test to handling losses, compositing of samples to arrive at an average vapor pressure for the contents of large vessels shall not be permitted.

(*a*) *Sampling into Open-Type Sample Containers.*—Open-type sample containers (Section 9) shall be used for obtaining samples from large open tanks and may be used for obtaining samples from closed or pressure tanks. In both cases the containers shall be rinsed with the product to be sampled immediately prior to obtaining the sample. The container shall then be refilled. If necessary, enough of the contents shall be poured off immediately so that the portion remaining shall fill the sample container to not less than 70 per cent and not more than 80 per cent of its capacity. The sample container shall be promptly sealed. The sampling from closed or pressure tanks shall be conducted under such conditions that the sample in the sample container is at a temperature of 32 to 40 F. for the duration of sampling. If this requires the use of the chilled sampling apparatus (Section 8 and Fig. 2), sufficient volume shall be wasted through the cooling coil to insure complete purging. During the sampling operation, the outlet valve (*A*, Fig. 2) shall be throttled so as to maintain a pressure in the chilling coil equivalent approximately to that in the vessel being sampled.

(*b*) *Sampling into Closed-Type Sample Containers.*—Closed-type sample containers (Section 9) may be used for obtaining samples from closed vessels. The inlet valve of the container shall be connected to the sampling line from the pressure vessel. During the sampling operation, the outlet valve of the container shall be throttled so as to maintain a pressure in the container equivalent approximately to that in the vessel being sampled. The sample shall be drawn by allowing the product to flow through the sampling system until a volume equal to at least twice that of the sample container has been passed. Valves shall then be closed and the container disconnected. Enough of the contents shall be withdrawn immediately so that the portion remaining shall fill the sample container to not less than 70 per cent and not more than 80 per cent of its capacity. Should the vapor pressure of the product be insufficient to force liquid from the container, it shall be permissible to open both valves to facilitate the withdrawal of the amount specified. The sample container shall be promptly sealed.

Care of Samples

13. Samples shall be put in a cool place as soon as possible after they have been obtained and held there until all tests have been completed. Samples in leaky containers shall not be considered for tests but shall be discarded and new samples obtained.

Saturation of Samples in Sample Containers

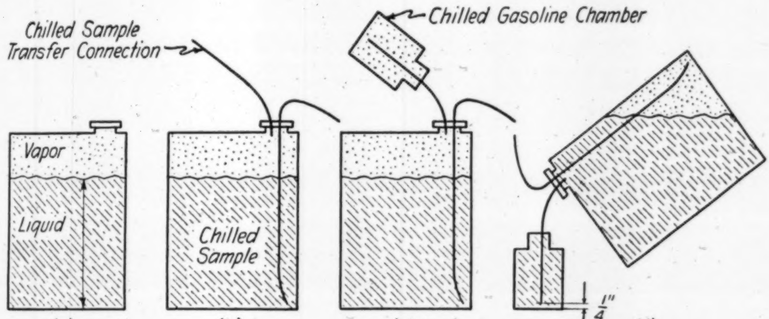
14. Prior to the Reid-vapor-pressure determinations, the sample container shall be immersed in the cooling bath (Section 4) held at a temperature of 32 to 40 F. for a sufficient time to allow the liquid to reach the bath temperature. The container shall then be unsealed for 5 sec., resealed, shaken vigorously, and then returned to the bath.

Sample Transfer

15. (*a*) The sample shall be transferred from the sample container to the gasoline chamber as described in the following Paragraphs (*b*) to (*d*). This operation shall be performed when the apparatus is being assembled for test and fits between the steps described in Section 17 (*d*) and (*e*).

(*b*) The gasoline chamber and the sample-transfer connection shall be chilled in the cooling bath (Section 4).

(*c*) With everything in readiness the chilled sample container shall be removed from the bath, the cap, stopper or plug shall be removed and the transfer connection immediately attached to the sample container. (See Fig. 3.)



(a) Sample Container Prior to Transfer of Sample

(b) Sealing Closure Replaced by Sample Transfer Connection

(c) Gasoline Chamber Placed Over Liquid Delivery Tube

(d) Position of System for Sample Transfer

FIG. 3.—Simplified Sketches Outlining Method of Transferring Sample to Gasoline Chamber from Open-Type Containers.

(*d*) The chilled gasoline chamber shall then be placed over the sample-delivery tube of the transfer connection. The entire system shall then be rapidly inverted so that the gasoline chamber is finally in an upright position with the delivery tube extending to within $\frac{1}{4}$ in. of the bottom of the gasoline chamber. In the case of closed-type containers the valves (or valve) shall not be opened until the system has reached its final position (Note). The gasoline chamber shall be filled to overflowing.

NOTE.—The valve on the sample-delivery tube should be opened first. If liquid does not flow, it will be necessary to open the second valve.

PROCEDURE

Preparation of Apparatus

16. If tests have been previously conducted in the apparatus to be used, the procedure prescribed in the following Paragraphs (*a*) to (*c*) shall be followed to prepare the apparatus for the next test. These operations are most conveniently done at the conclusion of each test.

(*a*) The air chamber, gasoline chamber and pressure gage shall be disconnected.

(*b*) The pressure gage shall be purged of residual gasoline by directing a small jet of air into its Bourdon tube for at least 5 min.

(*c*) The air chamber shall be thoroughly purged of liquid gasoline and gasoline vapors by filling the air chamber with warm water (above 90 F.) and allowing it to drain, repeating the operation at least five times (Note).

NOTE.—If this purging is done in a bath be sure to avoid small and unnoticeable films of floating gasoline by keeping the bottom and top openings of the air chamber closed as they pass through the surface of the water.

"Initial Air Temperature"; Assembly of Apparatus

17. (*a*) The air chamber shall be rinsed (see Note, Section 16 (*c*)) with clean water (temperature optional).

(*b*) The pressure gage shall then be attached to the air chamber.

(*c*) The air thermometer shall next be inserted in the air chamber for three-fourths of its length. The bulb shall not be permitted to rest on the wall of the air chamber.

(*d*) The temperature of the air and water vapor in the air chamber shall be read. Since it is absolutely necessary that this temperature shall be the actual temperature of the air and water vapor in the air chamber at the time of final assembly (Paragraph (*e*)), the air thermometer shall have been in the air chamber (Paragraph (*c*)) at least 5 min. before the temperature is read. This temperature shall be recorded as the "initial air temperature."

(*e*) The gasoline chamber, immediately (note exception in Section 22) after being filled with the sample (Section 15 (*b*), 21 (*b*) or (*c*)), shall be attached to the air chamber with the pressure gage attached (Paragraph (*b*)).

NOTE.—If a threaded coupling is employed a little water-soluble grease or other low-vapor-pressure lute on the threads will materially assist in making a tight connection.

(*f*) The time interval between the operations prescribed in Paragraphs (*d*) and (*e*) shall be as short as possible.

Vapor-Pressure Determination

18. (*a*) The assembled vapor-pressure bomb shall be turned upside down and shaken vigorously in this position. This operation shall be repeated several times.

(*b*) The bomb shall then be righted and immersed in the water bath to at least 1 in. above the top of the air chamber (Section 5).

(*c*) The water bath shall be maintained throughout the vapor-pressure determination at a constant temperature of 100 F. ±0.2 F. as determined by the bath thermometer (Section 6) well immersed in the bath.

(*d*) While the bomb is immersed in the bath, it shall be closely observed for leaks. Liquid leaks are much more difficult to detect than vapor leaks and since the much used coupling device is normally in the liquid section of the apparatus it shall be given particular attention. When, at any time in the course of the test, a leak is detected the test shall be discarded.

(*e*) After 5 min. the bomb shall be withdrawn from the bath, inverted, shaken vigorously and replaced in the bath. Thereafter, at intervals of not less than 2 min., the bomb shall be withdrawn from the bath, inverted, shaken vigorously and replaced in the bath. This vigorous shaking shall be performed quickly to avoid cooling the bomb and its contents.

(*f*) Prior to each removal of the bomb from the bath, the gage reading shall be observed. The gage shall be tapped lightly prior to each reading.

TABLE I.—CORRECTIONS TO CONVERT UNCORRECTED VAPOR PRESSURES TO REID VAPOR PRESSURES.

INITIAL AIR TEMPERATURE,[a] DEG. FAHR.	CORRECTIONS IN POUNDS PER SQUARE INCH FOR VARIOUS TEMPERATURES AS CALCULATED FOR NORMAL BAROMETRIC PRESSURES OF:				
	760 MM.	745 MM.	700 MM.	650 MM.	600 MM.
32	−2.9	−2.9	−2.7	−2.6	−2.5
40	−2.6	−2.6	−2.4	−2.3	−2.2
50	−2.2	−2.2	−2.1	−2.0	−1.9
60	−1.8	−1.8	−1.7	−1.6	−1.6
70	−1.4	−1.4	−1.3	−1.3	−1.2
80	−1.0	−1.0	−1.0	−0.9	−0.9
90	−0.5	−0.5	−0.5	−0.5	−0.5
100	0	0	0	0	0
110	+0.6	+0.6	+0.5	+0.5	+0.5

$$\text{Correction Factor} = \frac{(P_a - P_t)(t - 100)}{460 + t} - (P_{100} - P_t)$$

where t = the air chamber temperature at beginning of test, deg. Fahr.
P_t = the vapor pressure of water, pounds per square inch, at t deg. Fahr.
P_{100} = the vapor pressure of water, pounds per square inch, at 100 F.
P_a = the normal barometric pressure in pounds per square inch at place where test is conducted.

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[a] For intermediate air temperatures, the corrections should be interpolated to the nearest 0.1 lb. per sq. in.

(*g*) These operations shall be continued for at least 20 min. to insure equilibrium. After this time if consecutive gage readings observed are constant, the value of the readings shall be recorded as the "uncorrected vapor pressure" of the sample under test.

(*h*) The pressure gage shall be immediately removed from the bomb and its reading at the "uncorrected vapor pressure" compared with that of a mercury column. The correction thus determined shall be recorded as the "gage correction."

CALCULATION

Calculation

19. (*a*) To calculate the "Reid vapor pressure" of the sample under test, the "uncorrected vapor pressure" (Section 18 (*g*)) shall be corrected for the pressure-gage error (Section 18 (*h*)) and for the change in the pres-

sure of the water vapor and air in the air chamber due to the differences between the "initial air temperature" and the temperature of the water bath. Table I shows the corrections to apply for "initial air temperatures" ranging from 32 to 110 F. The value resulting from the application of these corrections shall be recorded as the "Reid vapor pressure."

Example.—If the "uncorrected vapor pressure" is 11.6 lb. and the mercury column reads 11.8 lb. when the gage reading is 11.6 lb. the "gage correction" is +0.2 lb. If the "initial air temperature" is 82 F. the correction (see Table I, Column 2) for air temperature is −0.9 lb. for Reid-vapor-pressure determinations at an atmospheric pressure of 745 mm. The combined correction is −0.7 lb. The "Reid vapor pressure" is 11.6 lb. minus 0.7 lb., or 10.9 lb.

(*b*) Because of the fact that the atmospheric pressure provided (Section 17 (*e*)) for in the air chamber counteracts the external atmospheric pressure, the only pressure acting internally on the pressure gage is the absolute vapor pressure of the liquid in the bomb so that the gage reading is the absolute vapor pressure of the liquid in pounds per square inch at 100 F. For purposes of brevity the "Reid vapor pressure" shall be recorded in pounds, without reference to temperature used in the test or to unit of surface.

REPRODUCIBILITY OF RESULTS

Reproducibility of Results

20. With proper attention to detail, duplicate tests for vapor pressure should not differ from each other by more than 0.2 lb. plus 1.0 per cent of the vapor pressure as determined.

SAMPLING PROCEDURE FOR PRODUCTS HAVING VAPOR PRESSURES OVER 26 LB.

The special sampling procedure described in the following Sections 21 and 22 is required because the evaporation losses inherent in the sampling procedure described in Sections 11 to 15, inclusive, affect the results substantially if applied to products having vapor pressures much above 26 lb., particularly if these products contain small proportions of very volatile components.

The complete method for products under this title comprises Sections 1 to 3, 5 to 7, 10, 21, 22, and 16 to 20, in the order listed.

21. (*a*) The sample shall be obtained as described in either of the following Paragraphs (*b*) or (*c*). This operation fits between the steps described in Section 17 (*d*) and (*e*).

(*b*) *Sampling by Immersion.*—The empty gasoline chamber shall first be immersed in the tank or container of the liquid to be tested and allowed to fill while being raised and lowered through the liquid. This operation shall be repeated. These first two fillings are for the purpose of thoroughly washing and chilling the chamber, which shall then be emptied. The chamber shall then be refilled in the same manner, care being exercised to see that it is completely filled with the liquid.

(c) *Sampling from Closed Vessels.*—The gasoline chamber equipped with valves, top and bottom (Fig. 1 (*b*)) shall be used. Before drawing the sample, the gasoline chamber shall be cooled to a temperature as low as or lower than that of the gasoline to be tested. The sample shall be taken as follows: Both valves shall first be closed and the gasoline chamber shall then be connected to the tank, pipe line, or other receptacle holding the liquid to be tested, by means of the lower valve of the chamber. The sample shall be taken at a place which will insure the obtaining of a representative sample of the product to be tested. With the upper valve closed, the valve on the container, tank or pipe line shall be opened and then the lower valve on the gasoline chamber shall be opened, allowing the full tank or line pressure to be exerted on the chamber. The upper valve on the chamber shall then be opened slightly and gasoline allowed to escape until a volume equal to at least twice the capacity of the gasoline chamber shall have been displaced. This procedure will insure the removal of all air from the chamber. Care shall be taken during the operations of purging and filling to prevent more than a slight drop in pressure between the chamber and the tank or line. After filling the gasoline chamber, both the top and bottom valves shall be closed in the order mentioned, and the chamber disconnected from the tank or pipe line (see Section 22).

Care of Samples

22. If samples are obtained in gasoline chambers of the type illustrated in Fig. 1 (*b*) and the air chamber is not immediately attached, both of the valves on the gasoline chamber shall be closed and plugged and the plugged gasoline chamber shall be kept chilled to a temperature lower than the temperature of the material sampled until the air chamber is attached (Section 17 (*e*)).

TENTATIVE METHODS OF TEST

FOR

KINEMATIC VISCOSITY[1]

A.S.T.M. Designation: D 445 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These methods are suitable for determining the kinematic viscosity of any petroleum product or lubricant which is a true viscous liquid at the temperature of the test.[2] Two procedures are included covering the use of the suspended-level viscosimeter and the modified Ostwald viscosimeter, respectively.

Method A.—Suspended-Level Viscosimeter[3]

Apparatus

2. The apparatus shall consist of one or more calibrated suspended-level viscosimeters, thermometers, bath, and timer conforming to the requirements specified in the following Paragraphs (*a*) to (*d*):

(*a*) *Suspended-Level Viscosimeters.*—The suspended-level viscosimeters (illustrated in Fig. 1 (*a*), (*b*) and (*c*)) shall be of glass and shall conform to the requirements in Table I. Instruments with one capillary are shown in Fig. 1 (*a*) and (*b*). In Fig. 1 (*c*) is shown an alternative form of viscosimeter in which two capillaries of different sizes are mounted in the same apparatus, which in effect increases the range available in a given bath.

All suspended-level viscosimeters shall be used only at efflux times greater than 80 sec. There is no upper limit for the efflux time but, in order to save time, it is suggested that a viscosimeter be selected for which the efflux time will be less than 1000 sec. Capillary diameter is not specified in Table I since it depends on the range of viscosities to be measured.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] These methods were developed in response to a need for a more scientific and accurate method for determining viscosity than by means of the Saybolt viscosimeter. Preliminary experiments indicate that the suspended level and modified Ostwald viscosimeters are subject to much smaller error than the Saybolt instrument. In addition, they measure kinematic viscosity and, therefore, the results are more directly translatable into friction, power requirement and heat development in bearings and other lubricated surfaces. Other forms of apparatus for determining kinematic viscosity are under consideration and may be added later.

[3] For further details of this method, reference should be made to the following articles:

Ogden FitzSimons, "A Rapid Precision Viscosimeter," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 7, p. 345 (1935).

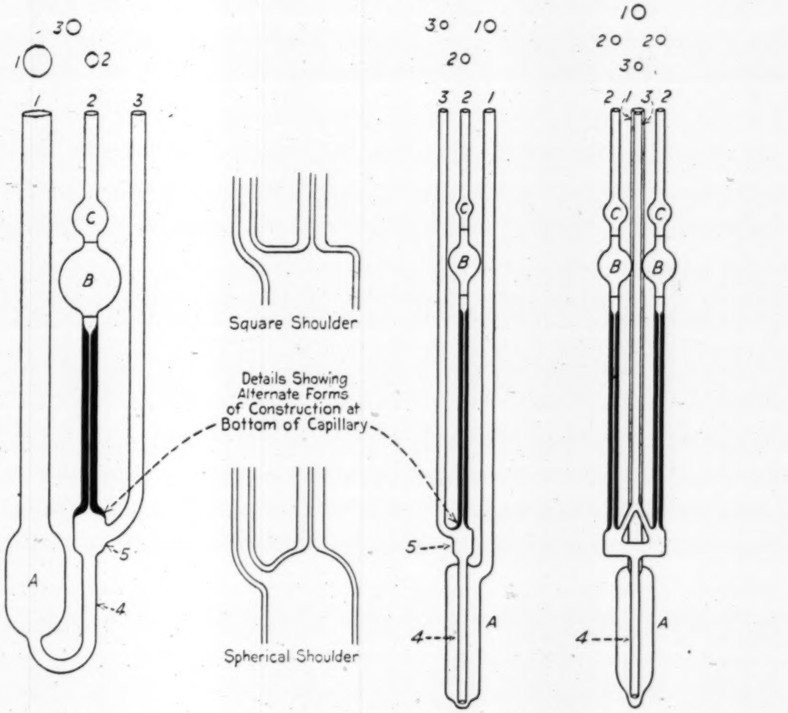
L. Ubbelohde, "The Simplest and Most Accurate Viscometer and Other Instruments with Suspended Level," *Journal*, Inst. Petroleum Technologists (London), Vol. 19, p. 376 (1933); also *Oel und Kohle*, Vol. 12, p. 949 (1936).

L. Ubbelohde, "The Suspended Level Viscometer," *Journal*, Inst. Petroleum Technologists (London), Vol. 22, p. 37 (1936).

L. Ubbelohde, "Viscosimeter," U. S. Patent No. 2048305, July 21, 1936.

L. Ubbelohde, "The Principle of the Suspended Level Applications to the Measurement of Viscosity." *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 9, p. 85 (1937).

L. Ubbelohde, "The Viscometer with Suspended Level (The Elimination of the Influence of Surface Tension by the Suspended Spheric Level)," *Journal*, Inst. Petroleum Technologists (London), Vol. 23, No. 165, p. 427 (1937).



(a) Ubbelohde Viscosimeter with One Capillary Tube.

(b) FitzSimons' Modification with One Capillary Tube.

(c) FitzSimons' Modification with Two Capillary Tubes.

FIG. 1.—Suspended-Level Viscosimeters.

For light oils it is necessary to use viscosimeters with smaller capillary diameters than for heavier oils. For viscosity ranges normally encountered, there are given in Table II suggested requirements for capillary diameters and calibration constants for five suspended-level viscosimeters (Note).

TABLE I.—DIMENSIONS OF SUSPENDED-LEVEL VISCOSIMETERS.

		UBBELOHDE VISCOSIMETER	FITZSIMONS' MODIFICATION
Over-all length, mm		260 to 290	300 to 340
Length of capillary, mm		85 to 95	115 to 125
Length from center of bulb *B* to bottom of capillary, mm		125 to 135	140 to 150
Volume between marks, ml		5.4 to 5.8	3.60 to 3.90
Outside diameter	of bulb *C*, mm	10 to 14	10 to 14
	of bulb *A*, mm	20 to 30	20 to 30
Length of bulb *A*, mm		40 to 50	70 to 80
Inside diameter	of tubes 1 and 4, mm	8 to 10	8 to 10
	of tubes 2 and 3, mm	3 to 5	4 to 5
	of tube 5 (square shoulder), mm	10 to 15	10 to 15
	of tube 5 (spherical shoulder), mm	17 to 20	14 to 17

Viscosimeter No. 1 is suitable for testing low-viscosity products such as kerosines and light fuel oils. The viscosimeters with larger capillary diameters (viscosimeters Nos. 1A to 5) cover the ranges of viscosities usually encountered with lubricating oils. It should be noted that the sizes shown in Table II are not mandatory; other capillary diameters may be used, provided the efflux time is greater than 80 sec.

NOTE.—In case the amount of testing does not warrant installation of five suspended-level viscosimeters it is possible, with equal accuracy, to make use of longer times of outflow, thereby eliminating one or more. For instance, in the case of the FitzSimons' modification, viscosimeter No. 2 may serve for viscosities varying from 3.0 to 30 centistokes and No. 4 from 20 to 1200 centistokes, thus making it unnecessary to install viscosimeters Nos. 3 and 5. A corresponding selection of instruments may also be made in the case of the Ubbelohde viscosimeter.

TABLE II.—SUGGESTED CAPILLARY DIAMETERS OF SUSPENDED-LEVEL VISCOSIMETERS WITH CORRESPONDING VISCOSITY RANGES AND CALIBRATION CONSTANTS.

Suspended-Level Viscosimeter	Efflux Time, sec.	Ubbelohde Viscosimeter				FitzSimons' Modification			
		Capillary Diameter, mm.	Approximate Calibration Constant, C^a	Approximate Viscosity Range		Capillary Diameter, mm.	Approximate Calibration Constant, C^a	Approximate Viscosity Range	
				Centistokes	Saybolt Universal Seconds			Centistokes	Saybolt Universal Seconds
No. 1........	80 to 1000	0.61 to 0.65	0.01	1 to 10	Up to 58	0.42 to 0.44	0.003	0.5 to 6.5	Up to 47
No. 1A......	80 to 1000	0.92 to 0.98	0.05	4 to 50	40 to 230	....	.....	......	......
No. 2........	80 to 1000	1.10 to 1.16	0.10	8 to 100	50 to 460	0.78 to 0.84	0.035	3.0 to 30	36 to 140
No. 3........	80 to 1000	1.96 to 2.04	1.0	80 to 1000	370 to 4600	1.02 to 1.08	0.10	9 to 100	55 to 460
No. 4........	80 to 1000	3.50 to 3.65	10.0	800 to 10 000	3700 to 46 000	1.28 to 1.36	0.25	20 to 250	100 to 115
No. 5........	80 to 1000	....	.....	....	....	1.92 to 2.00	1.2	100 to 1200	460 to 550

[a] The correct calibration constant depends on the actual dimensions of the instruments and therefore must be determined by experiment. The values in the above table are intended to serve as a guide only.

(*b*) The A.S.T.M. Kinematic Viscosity Test Thermometer shall conform to the following requirements. These specifications cover three total-immersion thermometers graduated in Fahrenheit degrees the ranges, as specified, being 97.5 to 102.5 F.; 127.5 to 132.5 F.; and 207.5 to 212.5 F., respectively.

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGE AND SUBDIVISIONS:
Range 97.5 to 102.5 F. subdivision in 0.1 F. for tests at 100 F.
Range 127.5 to 132.5 F. subdivision in 0.1 F. for tests at 130 F.
Range 207.5 to 212.5 F. subdivision in 0.1 F. for tests at 210 F.

TOTAL LENGTH: 235 to 241 mm. (9.25 to 9.48 in.).

STEM: Plain front, enamel back, suitable thermometer tubing. Diameter, 6.5 to 7.5 mm. (0.256 to 0.295 in.).

BULB: Corning normal or equally suitable thermometer glass.
Length, 45 to 55 mm. (1.77 to 2.17 in.).
Diameter, 6 mm. (0.236 in.) to not greater than stem.

ICE POINT: Graduated 31.5 to 32.5 F. in 0.1 F. division; 32 F. to be 80 to 85 mm. (3.15 to 3.35 in.) from bottom of bulb.

CONTRACTION CHAMBER: To be long narrow type, bottom not less than 100 mm. (3.94 in.) from bottom of bulb; top to be not more than 125 mm. (4.95 in.) from bottom of bulb.

DISTANCE TO LOWEST SPECIFIED GRADUATION FROM BULB: 140 to 155 mm. (5.51 to 6.10 in.).

DISTANCE TO HIGHEST SPECIFIED GRADUATION FROM TOP OF THERMOMETER: 25 to 40 mm. (98 to 1.57 in.).

EXPANSION CHAMBER: To permit heating to 220 F.

FILLING ABOVE MERCURY: Nitrogen gas.

TOP FINISH: Glass ring.

GRADUATIONS: All lines and figures clear cut and distinct. The first and each succeeding 0.5 F. line to be longer than the remaining lines. Graduations to be numbered at each 1 F.

IMMERSION: Total.

SPECIAL MARKINGS: "A.S.T.M. Kin. Vis.," a serial number and manufacturer's name or trade-mark shall be etched on the stem.

SCALE ERROR: The error at the viscosity point (100, 130, or 210 F.) not to exceed 0.10 F.

STANDARDIZATION: The thermometers are to be standardized at the ice point, and at the viscosity point (100, 130, or 210 F.) for conditions of total immersion, expressed to the nearest 0.02 F. and such corrections are to be added to the observed reading.

CASE: Each thermometer shall be supplied in a suitable case on which shall appear the marking "A.S.T.M. Kinematic Viscosity Thermometer," and the range.

NOTE.—For the purpose of interpreting these specifications the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

(*c*) *Bath.*—A bath suitable for immersion of the viscosimeter to within 5 cm. of the top, with provision for visibility of the instrument and the thermometer, shall be provided. The viscosimeter may be sealed in as an integral part of the bath. The thermometer shall be mounted with the bulb near the center of the viscosimeter. The bath shall be regulated so that the variation in the thermometer reading does not exceed 0.05 F. (that is, ± 0.025 F.). Either a liquid bath with thermostatic regulation and stirrer or a vapor bath with pressure regulation is permissible, the following materials being satisfactory for the purpose indicated:

BATH TEMPERATURE	LIQUID BATH	VAPOR BATH
100 F. (37.78 C.)............	Water	Methylene chloride
210 F. (98.89 C.)............	White oil Ethylene glycol	Tertiary amyl alcohol

(*d*) *Timer.*—The stop watch or other timing device used shall be graduated in divisions of 0.2 sec. or less, and shall be accurate to within 0.1 per cent when tested over a 60-min. period (Note).

NOTE.—Electrical timing devices are permissible provided they are accurate and capable of being read to 0.2 sec. Timing devices actuated by synchronous motors shall be used only on electric circuits of controlled frequency.

Procedure

3. (*a*) The proper suspended-level viscosimeter, according to Table II, shall be selected. The viscosimeter shall be cleaned by rinsing twice with petroleum ether, benzol, or a narrow cut of naphtha. This solvent shall

then be removed by passing a current of clean, dry air through the viscosimeter and for this reason the solvent used must be of such volatility that it will be easily and completely removed under these conditions.

NOTE 1.—The viscosimeter may be left in place in the bath, in which case the oil from the previous determination shall be withdrawn by suction through tube 3, after which portions of naphtha shall be successively introduced into tube 1, drawn up into tube 2 several times and removed by suction through tube 3. The tubes shall then be dried of naphtha by passing air through them.

NOTE 2.—Periodically, or whenever the appearance of the viscosimeter indicates the need of it, the instrument shall be cleaned by use of chromic acid which shall be allowed to stand in the tube for several hours, followed by thorough rinsing first with distilled water, then with c.p. acetone and drying by air current.

(*b*) The viscosimeter shall be immersed in the bath to within 5 cm. of the top and adjusted to a vertical position. This may be accomplished by visual examination in two different vertical planes or by employing a small plumb bob consisting of a 1-cm. length of solder wire and a piece of silk thread attached to a perforated cork placed in the wide arm of the instrument. When properly aligned the plumb bob will not touch the walls.

(*c*) A small sample of oil (about 10 ml.) shall be filtered through a fritted (sintered) glass filter of medium porosity or a 100-mesh screen filter into a small beaker or bottle. The oil sample as well as the viscosimeter must be free from any solid particles, lint, etc. The oil sample shall be charged into tube 1 of the viscosimeter.

(*d*) The bath shall be maintained at the temperature of test within 0.05 F. (that is, ± 0.025 F.). The viscosimeter shall remain in the constant-temperature bath long enough to reach the bath temperature. The minimum time required is 5 min. for the 100 F. (37.78 C.) bath, and 10 min. for the 210 F. (98.89 C.) bath.

(*e*) After the sample has attained bath temperature, tube 3 shall be stoppered with the finger and the oil shall be drawn into tube 2 by suction to some point above the upper mark but not above the center of the small bulb. (When using the double instrument, the tube 2 not being used must be closed as well as tube 3.)

(*f*) The suction shall then be released and the finger removed from tube 3, allowing the oil to flow freely out of the capillary tube. The time in seconds required for the meniscus to pass from the upper to the lower mark shall be measured. If this efflux time is less than 80 sec., the next smaller viscosimeter shall be selected and the operation repeated. Determinations shall be repeated until two successive efflux times agree within 0.2 per cent, the average of these two determinations being used for calculating the kinematic viscosity.

Reporting Results

4. The kinematic viscosity in centistokes shall be calculated from the following equation:

$$V = Ct - \frac{B}{t} \qquad (1)$$

where V = the kinematic viscosity in centistokes,

C = the determined calibration constant for the instrument,

t = the efflux time in seconds, and

B = an experimental constant determined by the design of viscosimeter. For viscosimeters with capillary diameters of 0.75 mm. or larger, the value of this constant is 2.8 for the Ubbelohde design and 1.0 for the FitzSimons' design; in the case of viscosimeters having capillary diameters less than 0.75 mm., the value of the constant shall be determined in accordance with Section 5 (*b*).

NOTE.—For routine use a tabulation in which the kinematic viscosity is shown for integral efflux times over the range used shall be prepared for each instrument based on Eq. (1) and on the calibration constant for the instrument. Interpolation tables should also be shown.

Calibration

5. The suspended-level viscosimeters shall be calibrated in accordance with one of the methods described in the following Paragraphs (*a*) and (*b*). The calibration constant determined at 100 F. (37.78 C.) shall be valid for use at other temperatures also.

(*a*) *For Viscosimeter with Capillary Diameter 0.75 mm. or Larger.*—The efflux time of an oil standard obtained from the American Petroleum Institute or National Bureau of Standards may be determined in the viscosimeter being calibrated. The efflux time for the oil standard shall be not less than 150 sec. The calibration constant, C, may then be calculated by substituting in Eq. (1) this efflux time and the kinematic viscosity of the oil standard and solving for the value of C.

An alternative procedure is to determine with the viscosimeter being calibrated the efflux time of an oil sample whose viscosity has been previously determined in a viscosimeter calibrated in accordance with Paragraph (*b*), or in a viscosimeter calibrated with a reference oil, or in a large viscosimeter calibrated as outlined in the reference appearing in the footnote to the title of Method B.[1] The calibration constant, C, may then be calculated by substituting in Eq. (1) this efflux time and the kinematic viscosity of the oil sample.

(*b*) *For Viscosimeter with Capillary Diameter Less than 0.75 mm.*—The constants B and C in Eq. (1) shall be determined for each viscosimeter, as follows: The efflux times of pure distilled water shall be determined at 68 F. (20 C.), 100 F. (37.78 C.), and 130 F. (54.44 C.), in the viscosimeter being calibrated. The temperature shall be maintained within ±0.02 F. (0.01 C.) of the desired test temperature. The kinematic viscosity of water shall be taken as 1.007 centistokes at 68 F. (20 C.), 0.689 centistokes at 100 F. (37.78 C.), and 0.518 centistokes at 130 F. (54.44 C.). The constants B and C_w (water) shall be the average of the three results calculated for each pair of temperatures from the following equations:

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[1] See p. 906.

$$B = \frac{t_1 t_2}{t_2^2 - t_1^2}(V_2 t_1 - V_1 t_2)$$

$$C_w = \frac{V_2 t_2 - V_1 t_1}{t_2^2 - t_1^2}$$

where B = the experimental constant determined by the design of the instrument,

C_w = the calibration constant determined with water as the calibrating liquid,

V_1 = the kinematic viscosity of water giving an efflux time of t_1, and

V_2 = the kinematic viscosity of water giving an efflux time of t_2.

The mean effective head does not affect the kinetic energy correction and accordingly, constant B is the same for all liquids. In the case of the viscosimeter with square-shouldered outlet tube, the mean effective head varies with liquids of different surface tension. It is accordingly necessary to compensate for the marked difference in surface tension of water and oil by multiplying C_w by 1.0035. This latter value is determined from the surface tensions of water and oil and is also based on the design of the instrument. The equation of the viscosimeter calibrated with water becomes as follows when used with petroleum fractions:

$$V = 1.0035\, C_w t - \frac{B}{t}$$

If a viscosimeter with a spherical shoulder is used, the head does not vary with surface tension; therefore, the multiplying constant is 1.000 instead of 1.0035.

Method B.—Modified Ostwald Viscosimeter[1]

Apparatus

6. The apparatus shall consist of one or more calibrated modified Ostwald viscosimeters, thermometers, bath, and timer conforming to the requirements specified in the following Paragraphs (*a*) to (*d*):

(*a*) *Modified Ostwald Viscosimeters.*—The modified Ostwald viscosimeters (illustrated in Fig. 2) shall be of glass and shall conform to the following dimensional requirements:

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[1] For further details of this method, reference should be made to "Viscosity Standardization of Petroleum Lubricating Fractions" by M. R. Cannon and M. R. Fenske, *Oil and Gas Journal*, Vol. 33, p. 52, April 11 (1935); and "Simple Viscometers and Auxiliary Equipment for the Accurate Measurement of Viscosity in Fundamental Units" by M. R. Cannon and M. R. Fenske, *Oil and Gas Journal*, Vol. 34, p. 45, April 9 (1936).

TABLE III.—DIMENSIONS OF MODIFIED OSTWALD VISCOSIMETERS.

Over-all length, mm		240 to 260
Length of capillary, mm		70 to 75
Volume of bulbs *A* and *B*, each, ml		3.0 to 3.3
Distance from center of bulb *B* to center of bulb *C*, mm		85 to 95
Inside diameter	of bulb *C*, mm	28 to 30
	of tube 1, mm	8 to 12
	of tube 2, mm	1.5 to 2.5
	of tube 3, mm	3 to 5

Each modified Ostwald viscosimeter shall be so constructed that a line drawn vertically through the center of bulbs *A* and *B* will pass through the center of bulb *C* (Fig. 2). Capillary diameter is not specified in Table III, since it depends on the range of the viscosity to be measured. For light oils it is necessary to use viscosimeters with smaller capillary diameters than for heavier oils. For the viscosity range normally encountered, there are given in Table IV suggested requirements for capillary diameters for four modified Ostwald viscosimeters (Note). It should be noted that the sizes shown in Table IV are not mandatory; other capillary diameters may be used provided the time of efflux is greater than 300 sec. for viscosimeters with capillary diameters less than 0.50 mm., 200 sec. for capillary diameters between 0.50 and 0.75 mm., and 100 sec. for capillary diameters greater than 0.75 mm.

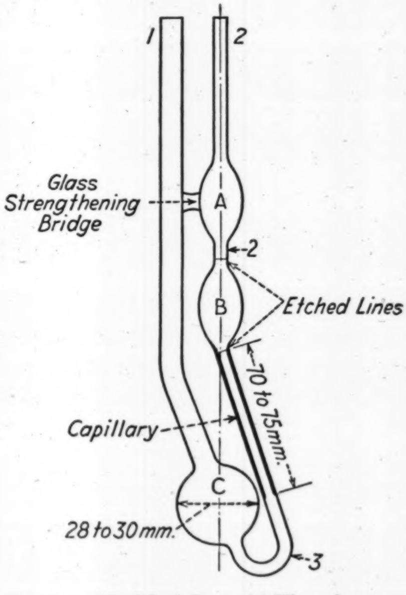

FIG. 2.—Modified Ostwald Viscosimeter.

NOTE.—In case the amount of testing does not warrant installation of four modified Ostwald viscosimeters it is possible, with equal accuracy, to make use of longer times of outflow, thereby eliminating one or more. For instance, viscosimeter No. 100 may serve for viscosities varying from 3 to 25 centistokes and No. 300 for viscosities from 25 to 850 centistokes, thus making it unnecessary to install viscosimeters Nos. 200 and 400.

(*b*) *Thermometers.*—As described in Section 2 (*b*).

(*c*) *Bath.*—As described in Section 2 (*c*).

(*d*) *Timer.*—As described in Section 2 (*d*).

Procedure

7. (*a*) The proper modified Ostwald viscosimeter, according to Table IV, shall be selected. The viscosimeter shall be cleaned by rinsing twice with petroleum ether, benzol, or a narrow cut of naphtha. This solvent shall then be removed by passing a current of air through the viscosimeter and for this reason the solvent used must be of such volatility that it will be easily and completely removed under these conditions.

(*b*) A small sample of oil (about 10 ml.) shall be filtered through a fritted (sintered) glass filter of medium porosity or a 100-mesh screen filter into a small beaker or bottle. The oil sample as well as the viscosimeter must be free from any solid particles, lint, etc.

TABLE IV.—SUGGESTED CAPILLARY DIAMETERS OF MODIFIED OSTWALD VISCOSIMETERS WITH CORRESPONDING VISCOSITY RANGES.

MODIFIED OSTWALD VISCOSIMETER	EFFLUX TIME, SEC.	CAPILLARY DIAMETER, MM.	APPROXIMATE VISCOSITY RANGE	
			CENTISTOKES	SAYBOLT UNIVERSAL SECONDS
No. 50	300 to 1500	0.40 to 0.45	0.8 to 3	......
No. 100	200 to 700	0.60 to 0.65	3 to 10	35 to 65
No. 200	100 to 700	0.97 to 1.03	10 to 70	60 to 325
No. 300	100 to 700	1.20 to 1.30	25 to 175	120 to 800
No. 400	100 to 700	1.80 to 1.90	120 to 850	550 to 4000
No. 500	100 to 700	2.70 to 2.90	800 to 5600	3600 to 25 000

(*c*) The oil at a temperature of 70 to 90 F. (21.11 to 32.22 C.) shall be charged into the viscosimeter in the following manner: The instrument shall be held in an inverted vertical position with the end of the capillary arm immersed in the oil. Both bulbs on the capillary side of the viscosimeter shall be filled with oil by suction, and the oil brought into the working capillary up to the mark etched on it. Care shall be used that the rubber tubing employed in applying the suction shall be clean.

(*d*) After loading, the excess oil shall be wiped off the top of the viscosimeter and it shall then be placed in the constant temperature bath. It shall be immersed in the bath liquid to within 5 cm. of the top.

NOTE.—A convenient way to support the viscosimeter in the bath is to clamp a rubber stopper on the instrument and to provide the cover of the constant-temperature bath with holes to receive the rubber stopper. A No. 10 rubber stopper with a 1.0-cm. hole and with a section 0.5 cm. in width cut out from the opening to the side of the stopper is suggested.

(*e*) The viscosimeter shall be adjusted to a vertical position. This may be accomplished by visual examination in two different vertical planes or by employing a small plumb-bob consisting of a 1-cm. length of solder wire and a piece of silk thread attached to a perforated cork placed in the wide arm of the instrument. When properly aligned the plumb bob will not touch the walls.

(*f*) The viscosimeter shall remain in the constant-temperature bath long enough to reach the bath temperature. The minimum time required is 5 min. for the 100 F. (37.78 C.) bath, and 10 min. for the 210 F. (98.89 C.) bath.

(*g*) During the heating stage the oil will drain from the upper bulbs into the lower reservoir and in the course of this operation a bubble of air may be trapped in the bend at the bottom of the instrument. When the oil level is brought to the etched line between the bulbs this air shall be removed, so that there will be an unbroken column of oil extending from the lower reservoir through the capillary during the test.

(*h*) After the sample has attained bath temperature, suction shall be applied to the capillary arm to draw the oil sample up to a point about 5 mm. above the mark between the bulbs. The suction shall then be released thereby opening this arm to the atmosphere. The time for the meniscus to pass from the upper to the lower mark shall be measured.

(*i*) The viscosity of the oil in centistokes shall then be obtained by multiplying the efflux time by the viscosimeter constant.

(*j*) A check determination shall be made by again drawing the oil sample above the etched line between the bulbs and timing as before. In careful work it may be desirable to run the same oil in two different viscosimeters.

NOTE.—An experienced operator can run six or more viscosimeters at one time. During the preheating stage of one viscosimeter, another instrument may be loaded or cleaned so that no time is lost. Small (10-in. diameter) constant-temperature baths will hold six or more viscosimeters.

Reporting Results

8. The kinematic viscosity in centistokes shall be calculated from the following equation:

$$V = Ct \qquad (2)$$

where V = the kinematic viscosity in centistokes,
C = the determined calibration constant for the instrument, and
t = the efflux time in seconds.

Calibration

9. The modified Ostwald viscosimeters shall be calibrated in accordance with one of the methods described in the following Paragraphs (*a*) and (*b*):

(*a*) The outflow time of an oil standard obtained from the American Petroleum Institute or National Bureau of Standards may be determined in the viscosimeter being calibrated. The efflux time for the oil standard shall be not less than 150 sec. The calibration constant C, may then be calculated by substituting in Eq. (2) this efflux time and the kinematic viscosity of the oil standard and solving for the value of C.

(*b*) An alternative procedure is to determine the calibration constant C by means of a master viscosimeter or calibrated instrument of the type as outlined in the reference appearing in the footnote to the title of Method B.[1]

(*c*) If it is desired to perform all calibrations at 100 F. (37.78 C.) the calibration constants at 60 F. (15.56 C.), 130 F. (54.44 C.), and 210 F. (98.89 C.) may be obtained, as follows:[2]

Calibration constant at 210 F. (98.89 C.) = 0.996 × calibration constant at 100 F. (37.78 C.)

Calibration constant at 130 F. (54.44 C.) = 0.999 × calibration constant at 100 F. (37.78 C.)

Calibration constant at 60 F. (15.56 C.) = 1.001 × calibration constant at 100 F. (37.78 C.)

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[1] See p. 906.

[2] A more general equation which exrpesses the change in the calibration constant of the modified Ostwald viscosimeter with variation in temperature is as follows:

$$T_2 = C_{T_1}\left[1 - \frac{\text{Vol.}_2 - \text{Vol.}_1}{0.785\, H\, d^2}\right]$$

where C_{T_2} = the calibration constant at T_2, the temperature for which the constant is being calculated,
C_{T_1} = the calibration constant at T_1, the temperature at which the constant is known,
Vol._2 = the total volume of liquid in the viscosimeter at T_2,
Vol._1 = the total volume of liquid in the viscosimeter at T_1,
H = the fluid head; for viscosimeters shown in Fig. 2, this is approximately 90 mm., and
d = the inside diameter of the lower reservoir; for viscosimeters shown in Fig. 2, this is approximately 30 mm.

TENTATIVE METHOD

FOR

CONVERSION OF KINEMATIC VISCOSITY TO SAYBOLT UNIVERSAL VISCOSITY[1]

A.S.T.M. Designation: D 446 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1937.

Scope

1. The conversion table and equation included in this method provide a means for converting kinematic viscosity in centistokes at any temperature to Saybolt Universal viscosity in seconds at the same temperature.[2,3]

Description

2. The Saybolt Universal viscosity equivalent to a given kinematic viscosity varies with the temperature at which the determination is made. The basic conversion values are those given in Table I for 100 F. The Saybolt Universal viscosity equivalent to a given kinematic viscosity at any temperature may be calculated as described in Section 3. Equivalent values at temperatures of 130 and 210 F. are given in Table I for convenience.

Procedure

3. For conversions at 100, 130 and 210 F., the equivalent Saybolt Universal viscosity values may be read directly from Table I. For kinematic viscosities between 2 and 70 centistokes which are not listed in Table I, the equivalent Saybolt Universal viscosity values may be obtained by linear interpolation. For kinematic viscosities over 70 centistokes, the Saybolt Universal viscosity equivalent may be calculated by use of the multiplication constant given in Table I corresponding to the proper temperature. The Saybolt Universal viscosity equivalent to a kinematic viscosity determined at t F. may be calculated by multiplying the equivalent Saybolt Universal viscosity at 100 F. by $1 + (t - 100)\ 0.000064$.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] See paper by C. E. Headington and J. C. Geniesse, "Method for Converting Kinematic Viscosity to Saybolt Universal Viscosity," p. 380.

[3] Extensive tables for converting centistokes to Saybolt Universal seconds are under preparation and when completed will be available from Society Headquarters, 260 S. Broad St., Philadelphia, Pa.

TABLE I.—VALUES FOR CONVERTING KINEMATIC VISCOSITY TO SAYBOLT UNIVERSAL VISCOSITY.

Kinematic Viscosity, Centistokes	Equivalent Saybolt Universal Viscosity in Seconds at Temperature of		
	100 F. (Basic Values, see Note)	130 F.	210 F.
2	32.6	32.7	32.8
2.5	34.4	34.5	34.6
3	36.0	36.1	36.3
3.5	37.6	37.7	37.9
4	39.1	39.2	39.4
4.5	40.7	40.8	41.0
5	42.3	42.4	42.6
6	45.5	45.6	45.8
7	48.7	48.8	49.0
8	52.0	52.1	52.4
9	55.4	55.5	55.8
10	58.8	58.9	59.2
11	62.3	62.4	62.7
12	65.9	66.0	66.4
13	69.6	69.7	70.1
14	73.4	73.5	73.9
15	77.2	77.3	77.7
16	81.1	81.3	81.7
17	85.1	85.3	85.7
18	89.2	89.4	89.8
19	93.3	93.5	94.0
20	97.5	97.7	98.2
21	101.7	101.9	102.4
22	106.0	106.2	106.7
23	110.3	110.5	111.1
24	114.6	114.8	115.4
25	118.9	119.1	119.7
26	123.3	123.5	124.2
27	127.7	127.9	128.6
28	132.1	132.4	133.0
29	136.5	136.8	137.5
30	140.9	141.2	141.9
31	145.3	145.6	146.3
32	149.7	150.0	150.7
33	154.2	154.5	155.3
34	158.7	159.0	159.8
35	163.2	163.5	164.3
36	167.7	168.0	168.9
37	172.2	172.5	173.4
38	176.7	177.0	177.9
39	181.2	181.5	182.5
40	185.7	186.1	187.0
41	190.2	190.6	191.5
42	194.7	195.1	196.1
43	199.2	199.6	200.6
44	203.8	204.2	205.2
45	208.4	208.8	209.9
46	213.0	213.4	214.5
47	217.6	218.0	219.1
48	222.2	222.6	223.8
49	226.8	227.2	228.4
50	231.4	231.8	233.0
55	254.4	254.9	256.2
60	277.4	277.9	279.3
65	300.4	301.0	302.5
70	323.4	324.0	325.7
Over 70	Saybolt seconds = centistokes × 4.620	Saybolt seconds = centistokes × 4.629	Saybolt seconds = centistokes × 4.652

NOTE.—To obtain the Saybolt Universal viscosity equivalent to a kinematic viscosity determined at *t* F. multiply the equivalent Saybolt Universal viscosity at 100 F. by 1 + (*t* – 100) 0.000064, for example, 10 centistokes at 210 F. are equivalent to 58.8 × 1.0070 or 59.2 Saybolt Universal seconds at 210 F.

Supplementary Conversion Equivalents

4. The following equivalents are frequently used in connection with viscosity conversions:

Poise = c. g. s. unit of absolute viscosity
Centipoise = 0.01 poise
Stoke = c. g. s. unit of kinematic viscosity
Centistoke = 0.01 stoke
Centipoises = centistokes × density (at temperature under consideration)
Reyn (1 lb. sec. per sq. in.) = 69×10^5 centipoises

Approved as
AMERICAN TENTATIVE STANDARD
by the American Standards Association
A.S.A. No.: Z11.39-1937

TENTATIVE VISCOSITY-TEMPERATURE CHARTS
FOR
LIQUID PETROLEUM PRODUCTS[1]

A.S.T.M. Designation: D 341 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1932; Revised, 1937.

Scope

1. The viscosity-temperature charts[2] covered by this standard are a convenient means for ascertaining the viscosity of a petroleum oil[3] at any temperature, within a limited range, provided viscosities at two temperatures are known. Conversely, the charts may be used to ascertain the temperature at which a desired viscosity is attained.

Precaution

2. The charts should be used only in that temperature range in which the petroleum oil is a liquid. A safe range is that between initial boiling point and cloud point. Few data are available for temperatures above 300 F.; therefore, it is probable that in this range the charts will not give as accurate results as at the lower temperatures.

Description

3. The charts are so constructed that for any given petroleum oil the viscosity-temperature points lie on a straight line. Four charts are available, as follows:

Chart A. Saybolt Universal Viscosity:
Viscosity: 33 to 100,000,000 Saybolt Universal seconds
Temperature: –30 to +450 F.
Size: 0 by 1 6 in.

Chart B. Saybolt Universal Viscosity, Abridged:
Viscosity: 33 to 100,000 Saybolt Universal seconds
Temperature: –30 to +350 F.
Size: 8½ by 11 in.

Chart C. Kinematic Viscosity, High Range:
Viscosity: 2 to 20,000,000 centistokes
Temperature: –30 to +450 F.
Size: 20 by 16 in.

Chart D. Kinematic Viscosity, Low Range:
Viscosity: 0.40 to 100 centistokes
Temperature: –30 to +450 F.
Size: 20 by 20 in.

Charts A and B have Saybolt Universal viscosity lines only, whereas Charts C and D have kinematic viscosity lines only. Charts C and D are on the same scale so that they may be superimposed on one another

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[1] Under the standardization procedure of the Society, these charts are under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Facsimiles of two of the charts are given in Figs. 1 and 2.

[3] While the charts are based on the behavior of petroleum products, information as to their applicability to other liquids is earnestly solicited and should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 50 W. 50th St., New York City.

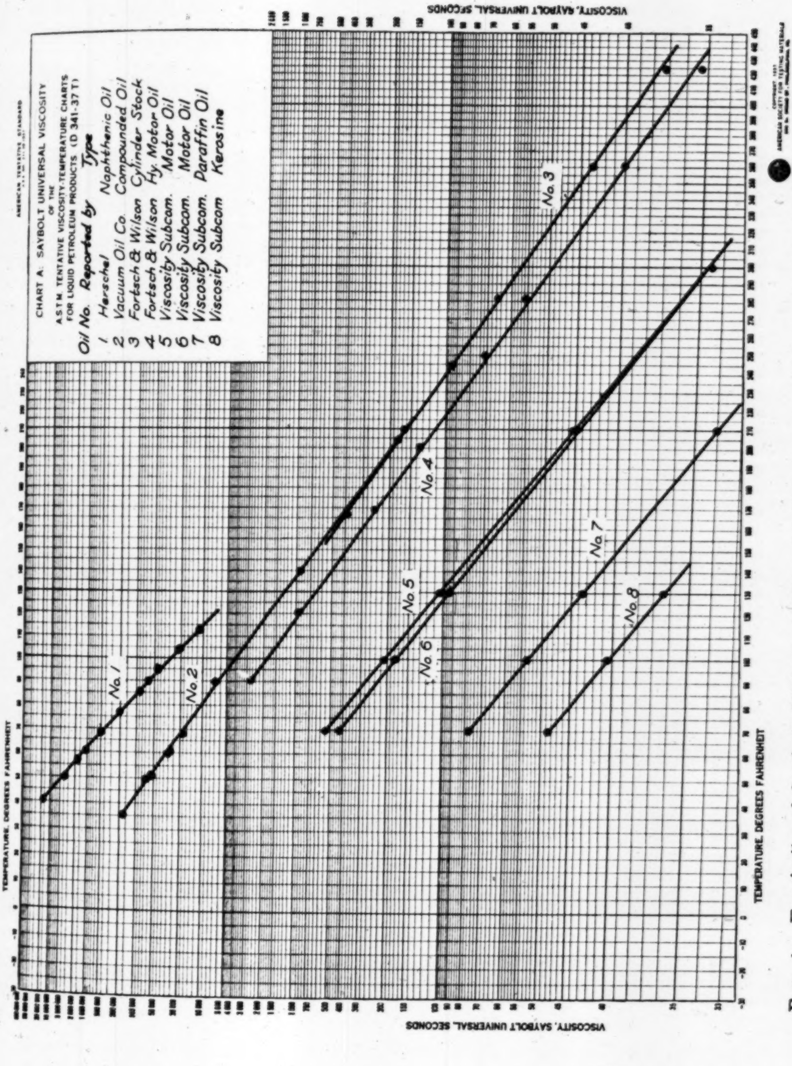

FIG. 1.—Facsimile of Saybolt Universal Viscosity-Temperature Chart A on Which Several Typical Experimental Curves Have Been Plotted (Chart Size: 20 by 16 in.).

over the range 2 to 100 centistokes to make one continuous chart from 0.40 to 20,000,000 centistokes. Chart A is also on the same scale. The ranges covered by Chart B are those most generally used in viscosity determinations and this portion of Chart A has been reproduced on a

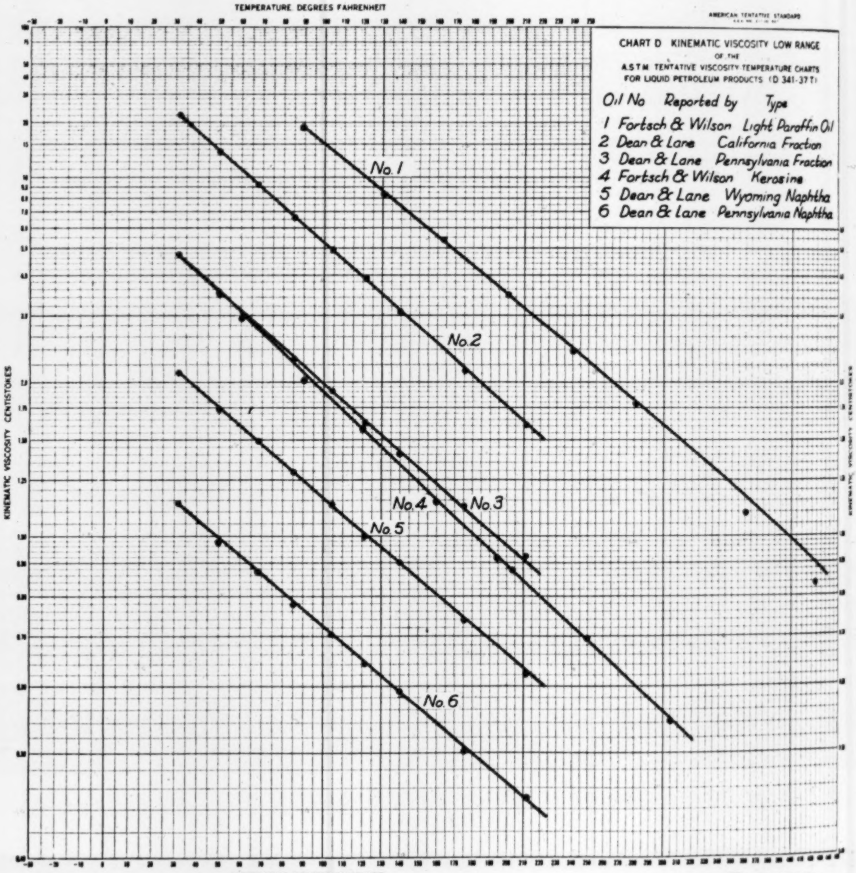

FIG 2.—Facsimile of Kinematic Viscosity-Temperature Chart D, Low Range, on Which Several Typical Experimental Curves Have Been Plotted (Chart Size: 20 by 20 in.).

slightly reduced scale as a convenience for certain types of plotting where the accuracy of Chart A is not considered essential.

NOTE.—The Viscosity-Temperature Charts are available as follows:

Chart A.	Saybolt Viscosity (20 by 16 in.)	Single copy, 25 cents; pad of 25, \$1.50; four pads, \$5.00
Chart B.	Saybolt Viscosity, Abridged ($8\frac{1}{2}$ by 11 in.)	Pad of 50, \$2.00; three pads, \$5.00
Chart C.	Kinematic Viscosity, High Range (20 by 16 in.)	Single copy, 25 cents; pad of 25, \$1.50; four pads, \$5.00
Chart D.	Kinematic Viscosity, Low Range (20 by 20 in.)	Single copy, 25 cents; pad of 25, \$1.50; four pads, \$5.00

Procedure

4. Plot the two known viscosity-temperature points[1] on the chart. With great care draw accurately a sharply defined, straight line through them. A point on this line, within the range defined in Section 2, shows the viscosity at the corresponding desired temperature, and *vice versa*.

Extrapolation

5. Viscosity-temperature points on the extrapolated portion of the line, but still within the range defined in Section 2, are satisfactory provided the viscosity-temperature line is located quite accurately. For purpose of extrapolation it is especially important that the two known viscosity-temperature points be far apart. If these two viscosity-temperature points are not sufficiently far apart, experimental errors in the viscosity determinations and in drawing the line may seriously affect the accuracy of extrapolated points, particularly if the difference between an extrapolated temperature and the nearest temperature of determination is greater than the difference between the two temperatures of determination. In extreme cases a viscosity determination at a third temperature is advisable.

APPENDIX

In addition to their primary purpose, these viscosity-temperature charts furnish a convenient means for estimating the viscosity at a given temperature of blends of petroleum liquids, provided viscosities of the component liquids at the given temperature are known. For this purpose the 0 F. line may be assumed to represent 100 per cent of the light component and the 100 F. line to represent 100 per cent of the heavy component. Plot the known viscosities of the two components on the proper lines at points corresponding to the values on the viscosity scale, and draw a straight line through them. Points on this line show viscosities at the given temperature of blends of the particular components, depending upon the proportion of the light and heavy component.

Example.—As an example, estimate the viscosity of a blend of 15 per cent of an oil having a viscosity of 38 seconds, Saybolt Universal at 100 F. and 85 per cent of an oil having a viscosity of 153 seconds, Saybolt Universal at 100 F. Draw a straight line between the 38 seconds Saybolt point on the 0 F. line and the 153 seconds Saybolt point on the 100 F. line. The printed figures between 0 and 100 F. represent the percentage of the heavy component. The line crosses the 85 F. line at a value corresponding to 105 seconds. Thus the estimated viscosity of the blend is 105 seconds, Saybolt Universal at 100 F. The experimental value was 108 seconds.

NOTE.—In case one of the components is motor gasoline, plot the gasoline point on the extension of the 0 F. line 3.75 in. below its intersection with the 33 seconds Saybolt Universal line and plot the point corresponding to the heavy component on the 100 F. line. Points on the straight line drawn between these two points show viscosities of blends of motor gasoline and oil at the same temperature as the components.

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[1] If viscosities are not known they should be determined in accordance with the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88), 1936 Book of A.S.T.M. Standards, Part II, p. 982, or the Tentative Methods of Test for Kinematic Viscosity (A.S.T.M. Designation: D 445 - 37 T) of the American Society for Testing Materials, see p. 900.

Conversion of centistokes to Saybolt seconds may be obtained from the Tentative Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity (A.S.T.M. Designation: D 446 - 37 T) of the American Society for Testing Materials, see p. 911.

TENTATIVE SPECIFICATIONS

FOR

STANDARD SIZES OF COARSE AGGREGATES FOR HIGHWAY CONSTRUCTION[1]

A.S.T.M. Designation: D 448 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover standard size designations and maximum permissible ranges in mechanical analyses for standard sizes of coarse aggregates and screenings for use in the construction or maintenance of various types of highways and highway structures.

Manufacture

2. The standard sizes of coarse aggregates prescribed in these specifications may be manufactured by means of any suitable commercial process and by the use of any sizes or shapes of plant screen openings necessary to produce the designated sizes within the limits of the mechanical analyses specified in Section 3.

Standard Sizes

3. Standard sizes of coarse aggregates shall comply with the sizes given in Tables I and II for Groups A and B, respectively. All sizes shall be determined by means of laboratory sieves having square openings.

NOTE.—Two groups of standard sizes, Groups A and B, are specified to satisfy the requirements for various localities. A study of the two groups will show that they overlap and include apparent duplications. It is emphasized, however, that it is not intended that all or a portion of the sizes from both groups will be adopted for any one locality. In any marketing area it is expected that specifications will be based on either Group A or B, whichever is more suitable for local conditions. Each of the sizes is designated by a size number such as No. 1A or No. 12A, for which the corresponding nominal sizes and the permissible ranges in mechanical analyses are given. The combined or modified sizes can be made by a combination of certain primary sizes or by a modification of these sizes, and the size numbers indicate which of the primary sizes have been combined or modified. Thus No. 12A results from a combination of sizes Nos. 1A and 2A; likewise No. 4A (modified) results from a modification of size No. 4A.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

These specifications are in effect a revision and consolidation of, and replace the former Tentative Specifications for Commercial Sizes of Broken Stone and Broken Slag for Highway Construction (D 63 - 23 T) and for Commercial Sizes of Sand and Gravel for Highway Construction (D 64 - 20 T), which specifications were accordingly discontinued in 1937.

TABLE I.—GROUP A, STANDARD SIZES OF COARSE AGGREGATES.

Size Number	Nominal Size, Square Openings	Amounts Finer than Each Sieve (Square Openings), per cent by weight											
		4 in.	3½ in.	2½ in.	2 in.	1½ in.	1 in.	¾ in.	½ in.	⅜ in.	No. 4	No. 8	No. 100
PRIMARY SIZES													
No. 1A	2 to 3½ in.	100	90 to 100	......	0 to 15	......	......	......	......	......	......	......	......
No. 2A	1 to 2 in.	......	......	100	90 to 100	35 to 70	0 to 15	......	......	......	......	......	......
No. 3A	½ to 1 in.	......	......	......	......	100	90 to 100	......	0 to 15	......	......	......	......
No. 4A	No. 4 to ½ in.	......	......	......	......	......	......	100	90 to 100	40 to 75	0 to 15	0 to 5	......
No. 5A	0 to No. 4	......	......	......	......	......	......	......	100	......	75 to 100	......	10 to 30
COMBINED AND MODIFIED SIZES													
No. 12A	1 to 3½ in.	100	90 to 100	......	25 to 60	......	0 to 15	......	......	......	......	......	......
No. 23A	½ to 2 in.	......	......	100	90 to 100	......	20 to 55	......	0 to 15	......	0 to 5	......	......
No. 234A	No. 4 to 2 in.	......	......	100	95 to 100	......	35 to 70	......	10 to 30	......	0 to 5	......	......
No. 34A	No. 4 to 1 in.	......	......	......	......	100	90 to 100	......	25 to 60	......	0 to 10	......	......
No. 345A	0 to 1 in.	......	......	......	......	100	90 to 100	......	30 to 65	......	10 to 25	......	0 to 10
No. 45A	0 to ½ in.	......	......	......	......	......	......	100	90 to 100	......	......	......	0 to 20
No. 3A (Modified)	No. 4 to 1 in.	......	......	......	......	100	90 to 100	40 to 75	5 to 20	......	0 to 5	......	......
No. 4A (Modified)	No. 8 to ½ in.	......	......	......	......	......	......	100	90 to 100	40 to 70	5 to 25	0 to 5	......
No. 5B (Modified)	No. 8 to ⅜ in.	......	......	......	......	......	......	......	100	90 to 100	10 to 30	0 to 8	......

TABLE II.—GROUP B, STANDARD SIZES OF COARSE AGGREGATES.

Size Number	Nominal Size, Square Openings	Amounts Finer than each Sieve (Square Openings), per cent by weight												
		4 in.	$3\frac{1}{2}$ in.	3 in.	$2\frac{1}{2}$ in.	2 in.	$1\frac{1}{2}$ in.	1 in.	$\frac{3}{4}$ in.	$\frac{1}{2}$ in.	$\frac{3}{8}$ in.	No. 4	No. 8	No. 100
PRIMARY SIZES														
No. 1B	$2\frac{1}{2}$ to $3\frac{1}{2}$ in.	100	90 to 100	35 to 70	0 to 15	...	...	...	...	...	...	...	...	...
No. 2B	$1\frac{1}{2}$ to $2\frac{1}{2}$ in.	...	...	100	90 to 100	35 to 70	0 to 15	...	...	...	...	...	...	...
No. 3B	$\frac{3}{4}$ to $1\frac{1}{2}$ in.	...	...	...	...	100	90 to 100	20 to 55	0 to 15	...	...	...	...	...
No. 4B	$\frac{3}{8}$ to $\frac{3}{4}$ in.	...	...	...	...	...	...	100	90 to 100	20 to 55	0 to 15	...	...	...
No. 5B	No. 4 to $\frac{3}{8}$ in.	...	...	...	...	...	...	...	...	100	90 to 100	0 to 20	0 to 5	...
No. 6B	0 to No. 4	...	...	...	...	...	...	...	...	...	100	85 to 100	...	10 to 30
COMBINED AND MODIFIED SIZES														
No. 12B	$1\frac{1}{2}$ to $3\frac{1}{2}$ in.	100	90 to 100	...	35 to 70	...	0 to 15	...	...	...	...	...	...	...
No. 23B	$\frac{3}{4}$ to $2\frac{1}{2}$ in.	...	...	100	90 to 100	...	25 to 60	...	0 to 10	...	...	...	...	...
No. 34B	$\frac{3}{8}$ to $1\frac{1}{2}$ in.	...	...	...	...	100	90 to 100	...	30 to 65	...	0 to 10	0 to 5	...	...
No. 3A (Modified)	No. 4 to 1 in.	...	...	...	...	...	100	90 to 100	40 to 75	5 to 20	...	0 to 5	...	...
No. 345B	No. 4 to $1\frac{1}{2}$ in.	...	...	...	...	100	95 to 100	...	35 to 70	...	10 to 30	0 to 5	...	...
No. 45B	No. 4 to $\frac{3}{4}$ in.	...	...	...	...	...	...	100	90 to 100	...	20 to 55	0 to 10	0 to 5	...
No. 456B	0 to $\frac{3}{4}$ in.	...	...	...	...	...	...	100	90 to 100	...	30 to 65	10 to 25	...	0 to 10
No. 56B	0 to $\frac{3}{8}$ in.	...	...	...	...	...	...	...	...	100	90 to 100	...	...	0 to 25
No. 5B (Modified)	No. 8 to $\frac{3}{8}$ in.	...	...	...	...	...	...	...	...	100	90 to 100	10 to 30	0 to 8	...
No. 45B (Modified)	No. 8 to $\frac{3}{4}$ in.	...	...	...	...	...	...	100	90 to 100	...	30 to 65	5 to 25	0 to 5	...

TENTATIVE METHOD OF TEST
FOR
ABRASION OF GRAVEL BY USE OF THE DEVAL MACHINE[1]

A.S.T.M. Designation: D 289 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1928; REVISED, 1937.

Scope

1. This method outlines a procedure for testing uncrushed or crushed gravel for resistance to abrasion in the Deval abrasion testing machine with an abrasive charge. Uncrushed gravel consists entirely of uncrushed fragments and, for the purpose of this test, a gravel containing more than 10 per cent by weight of crushed fragments shall be considered as crushed gravel.

Deval Machine

2. The Deval abrasion testing machine[2] shall be used. The machine shall consist of one or more hollow cast-iron cylinders; closed at one end and furnished with a tightly-fitting iron cover at the other. The inside dimensions of the cylinders shall be 20 cm. in diameter and 34 cm. in depth. These cylinders shall be mounted on a shaft at an angle of 30 deg. with the axis of rotation of the shaft.

Abrasive Charge

3. An abrasive charge weighing 2500 g. ± 10 g., comprising six cast-iron or steel spheres approximately $1\frac{7}{8}$ in. in diameter, each weighing between 405 and 440 g. shall be used with each test sample. The cast-iron spheres shall conform to the following requirements as to chemical composition:

Combined carbon, min., per cent	2.50
Graphitic carbon, max., per cent	0.25
Silicon, max., per cent	1.00
Manganese, max., per cent	0.50
Phosphorus, max., per cent	0.25
Sulfur, max., per cent	0.08

Grading

4. The gravel shall be separated by screening into the various sizes required for grading the test sample according to one of the gradings specified in Section 5. The material of these sizes shall be washed and dried.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] This machine is the same as specified in the Standard Method of Test for Abrasion of Rock by Use of the Deval Machine (A.S.T.M Designation: D 2), see 1936 Book of A.S.T.M. Standards, Part II, p. 1040.

Test Sample

5. (*a*) The test sample shall consist of dry gravel made up of percentages of the various sizes conforming to one of the gradings shown in the following table. The grading selected for the test sample shall be that most nearly representing the gravel furnished for the work.

Grading	Size of Square Openings in Inches (Standard Sieves) Passing	Size of Square Openings in Inches (Standard Sieves) Retained on	Percentage of Sample
A	¾-in.	½-in.	25
	1-in.	¾-in.	25
	1½-in.	1-in.	25
	2-in.	1½-in.	25
B	¾-in.	½-in.	25
	1-in.	¾-in.	25
	1½-in.	1-in.	50
C	¾-in.	½-in.	50
	1-in.	¾-in.	50
D	½-in.	¼-in.	50
	¾-in.	½-in.	50

(*b*) The weight of the test sample shall depend upon its average bulk specific gravity and shall be as follows:

Range in Bulk Specific Gravity	Weight of Sample, g.
Over 2.8	5500
2.4 to 2.8	5000
2.2 to 2.39	4500
Less than 2.2	4000

(*c*) When the gravel furnished for the work contains as much as 25 per cent of material finer than ½ in. but is of such size that either Grading *A*, *B*, or *C* would be used for the abrasion test, a second abrasion test shall be made using Grading *D*, if, in the opinion of the engineer, the particles less than ½ in. in size are not at least equal in hardness to those particles ½ in. or over in size.

Crushed Gravel

6. Gravel containing more than 10 per cent by weight of crushed fragments (Note) shall, for the purpose of this test, be considered as crushed gravel. In such cases, the test sample shall contain crushed fragments so as to be representative of the gravel furnished for the work and shall be prepared in accordance with Sections 4 and 5.

NOTE.—For the purpose of this test, a crushed gravel fragment may be considered as a fragment of gravel having at least one fractured face.

Procedure

7. The test sample and the abrasive charge shall be placed in the Deval abrasion testing machine and the cylinders shall be rotated for 10,000 revolutions at a speed of from 30 to 33 r.p.m.[1] At the completion of

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[1] These requirements are the same as those specified in the Standard Method of Test for Abrasion of Rock by Use of the Deval Machine (A.S.T.M. Designation: D 2), see 1936 Book of A.S.T.M. Standards, Part II, p. 1040.

the test, the material shall be removed from the machine and sieved on a No. 12 sieve conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[1] The material retained upon the sieve shall be washed, dried, and accurately weighed to the nearest gram.

Calculation

8. (*a*) *Uncrushed Gravel.*—In the case of uncrushed gravel, the loss by abrasion shall be considered as the difference between the original weight of the test sample and the weight of the material retained on the No. 12 sieve, expressed as a percentage of the original weight of the test sample.

(*b*) *Crushed Gravel.*—In the case of crushed gravel, the percentage by weight of crushed fragments shall be determined and the permissible percentage of wear which shall govern shall be calculated from the following formula:

$$W = \frac{A\,L + (100 - A)\,L'}{100}$$

where W = the permissible percentage of wear,

A = the percentage of uncrushed fragments,

$100 - A$ = the percentage of crushed fragments,

L = the maximum percentage of wear permitted by the specifications for gravel consisting entirely of uncrushed fragments, and

L' = the maximum percentage of wear permitted by the specifications for gravel consisting entirely of crushed fragments.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

TENTATIVE SPECIFICATIONS

FOR

ASPHALT CAP SHEET SURFACED WITH COARSE MINERAL GRANULES[1]

A.S.T.M. Designation: D 371 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1933; Revised, 1937.

Scope

1. These specifications cover asphalt roofing in sheet form, either 32 or 36 in. in width, composed of asphalt-saturated roofing felt coated on approximately one half of the width of the weather side with asphalt and the coated portion surfaced with mineral granules. This roofing is used as a cap sheet in the construction of built-up roofs.

Manufacture

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with a hot asphalt saturant, then coated on the weather side for approximately one half the width of the sheet with an asphaltic coating (compounded with a fine mineral filler substantially insoluble in water) and surfaced by embedding mineral granules in the hot asphaltic coating. The coated and surfaced portion shall be 15 in. in width on 32-in. roofing and 17 in. in width on 36-in. roofing.

Character of Felt

3. The felt shall be composed of a continuous sheet of "felted" vegetable and animal fibers. Its surface shall be uniformly smooth and free from lumps of unbeaten stock and particles of hard foreign substances.

Character of Saturant and Coatings

4. The saturant and coatings shall be composed principally of asphaltic materials.

Character of Felt After Saturation

5. The felt shall be thoroughly and uniformly saturated and shall show no unsaturated spots at any point upon cutting strips at random across the entire sheet and splitting them open for their full length.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

Properties

6. The fabricated product shall conform to the following requirements:

(a) Width		The roofing shall be put up in rolls and shall be either 32 or 36 in. in width, as may be specified, within a permissible variation of ±¼ in.	
(b) Area		The average area of the rolls examined shall contain sufficient roofing to cover 50 sq. ft. of roof surface, for roofing 32 in. in width, 107.3 sq. ft. ± 5 per cent, and for roofing 36 in. in width, 106.8 sq. ft. ± 5 per cent.	
		45-lb. Grade	**55-lb. Grade**
(c) Weight per roll, net average, lb		42	52
(d) Weight of dry felt per 108 sq. ft., lb		7.3	10.8
(e) Weight of saturant (soluble in carbon disulfide) per 108 sq. ft.[a]		11.0	17.5
(f) Weight of coating (soluble in carbon disulfide) per 108 sq. ft.[b]	min., lb.	12	12
	max., lb.	20	20
(g) Weight of mineral matter per 108 sq. ft. passing No. 6, retained on No. 100 sieve[b]	min. lb.,	25	25
	max., lb.	40	40
(h) Weight of mineral matter per 108 sq. ft. passing No. 100 sieve[b]	min., lb.	4	4
	max., lb.	10	10
(i) Weight of any 108 sq. ft. roll of complete roofing in shipment, min., lb		40	50
(j) Pliability at 77 F. (25 C.)[b]		At least 8 strips out of 10 from the granule-surfaced portion shall not crack when bent 90 deg. at a uniform speed over a rounded corner of ¾ in. in radius.	
(k) Behavior on heating to 176 F. (80 C.) for 2 hr.[b]		Not more than 1.5 per cent volatile matter loss. The granular surfacing shall not slide more than ¼ in. when suspended vertically.	

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[a] The weight of saturant per 108 sq. ft. shall not be less than 1.5 times the weight of the dry felt on the 45-lb. grade, and 1.6 times the weight of the dry felt on the 55-lb. grade.

[b] Granule-surfaced portion.

Surface Finish

7. (a) The surface of the weather side shall be uniform in finish and texture. The mineral granules on the surfaced portion shall be uniformly distributed in a smooth layer and shall be firmly embedded in the asphalt coating.

(b) The line between the surfaced and unsurfaced portion shall be straight and shall run parallel longitudinally to the edge of the sheet.

The surfaced portion shall be 15 in. ± ¼ in. on 32-in. roofing and 17 in. ± ¼ in. on 36-in. roofing.

(*c*) The reverse side of the sheet shall not be coated with asphalt, surfaced with mineral granules, or dusted with talc, mica or other fine mineral matter.

Freedom from Defects

8. The finished roofing shall be free from visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances and indentations.

Stickiness

9. The finished product shall not crack or be so sticky as to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50 F. (10 C.).

Packing

10. (*a*) Cap sheets shall be put up in rolls, containing sufficient material to cover not less than 50 sq. ft. No roll shall contain more than two pieces nor shall there be more than 3 per cent of rolls containing two pieces in any shipment. The rolls shall be tightly wound and securely wrapped in a substantial grade of paper completely encircling the roll and pasted at the overlap in such a manner as to prevent shifting from position. The ends of the rolls shall be protected with cloth covers or by other suitable means.

(*b*) Each roll shall be plainly marked with the manufacturer's name and brand. Special packing shall be agreed upon between the purchaser and the seller.

Inspection

11. Inspection of material shall be made as mutually agreed upon by the purchaser and the seller as part of the purchase contract.

Sampling and Testing

12. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (A.S.T.M. Designation: D 228 - 37 T) of the American Society of Testing Materials.[1]

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[1] See p. 957.

TENTATIVE SPECIFICATIONS

FOR

ASPHALT FOR DAMP-PROOFING AND WATERPROOFING[1]

A.S.T.M. Designation: D 449 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1917; ADOPTED IN AMENDED FORM, 1925;
REVERTED TO TENTATIVE, 1936; REVISED, 1937.

Scope

1. These specifications cover asphalt suitable for use as a mopping coat in damp-proofing, or as a plying or mopping cement in the construction of a membrane system of waterproofing.

Primer

2. The material used as a primer shall conform to the Tentative Specifications for Primer for Use with Asphalt in Damp-proofing and Waterproofing Below and Above Ground Level (A.S.T.M. Designation: D 41 – 36 T) of the American Society for Testing Materials[2] shall be used.

Membrane Waterproofing Materials

3. For the construction of a membrane system of waterproofing, any or all of the following felts or fabrics conforming to the specifications of the American Society for Testing Materials may be used alone or in various combinations:

(*a*) Standard Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (A.S.T.M. Designation: D 226).[3]

(*b*) Standard Specifications for Asphalt-Saturated Asbestos Felt for Use in Constructing Built-Up Roofs (A.S.T.M. Designation: D 250).[4]

(*c*) Standard Specifications for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (A.S.T.M. Designation: D 173),[5] asphalt type.

Types of Asphalts

4. The asphalts covered by these specifications are of two types, as follows:

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

These specifications are in effect a revision of, and replace the former Tentative Specifications for Asphalt for Use in Damp-proofing and Waterproofing Above Ground Level (D 144 – 36 T), and for Asphalt for Use in Damp-proofing and Waterproofing Below Ground Level (D 40 – 36 T), which specifications were accordingly discontinued in 1937.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 910 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 944.

[3] 1936 Book of A.S.T.M. Standards, Part II, p. 1137.

[4] *Ibid.*, p. 1120.

[5] *Ibid.*, p. 1134.

Type A.—A soft, adhesive "self-healing" asphalt which flows easily under the mop and which is suitable for use below ground level under uniformly moderate temperature conditions both during the process of installation and during service.

NOTE.—This type of asphalt is suitable for foundations, tunnels, subways, etc.

Type B.—A somewhat less susceptible asphalt with good adhesive and self-healing properties for use above ground level where not exposed to temperatures exceeding 125 F. (52 C.).

NOTE.—This type of asphalt is suitable for railroad bridges, culverts, retaining walls, tanks, dams, conduits, spray decks, etc.

Properties

5. The asphalt shall be homogeneous and free from water, and shall conform to the following requirements:

	TYPE A ASPHALT		TYPE B ASPHALT	
	MIN.	MAX.	MIN.	MAX.
(*a*) Softening Point (ring-and-ball method)	115 F. (46 C.)	145 F. (63 C.)	145 F. (63 C.)	170 F. (77 C.)
(*b*) Penetration:				
at 0 C. (32 F.), 200 g., 60 sec.	5	....	10	....
at 25 C. (77 F.), 100 g., 5 sec.	50	100	25	50
at 46 C. (115 F.), 50 g., 5 sec.	100	....	....	115
(*c*) Ductility at 25 C. (77 F.) (5 cm. per min.), cm	30	....	15	....
(*d*) Loss on heating at 163 C. (325 F.), 50 g., 5 hr., per cent.	....	2	....	1
(*e*) Penetration of residue, per cent original	60	....	60	....
(*f*) Bitumen soluble in carbon disulfide:				
Filled or native asphalt, per cent	95	....	....	....
Unfilled asphalt, per cent	99	....	99	....
(*g*) Bitumen soluble in carbon tetrachloride, per cent	99	....	99	....
(*h*) Ash:				
Filled or native asphalt, per cent.	....	5	....	....
Unfilled asphalt, per cent	....	1	....	1
(*i*) Coarse particles retained on No. 200 sieve as percentage of bitumen soluble in carbon disulfide, per cent	....	12	....	....
(*j*) Flash point (Cleveland open cup)	350 F. (175 C.)	....	400 F. (205 C.)	....

Methods of Sampling and Testing

6. The asphalt shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Standard Methods of Sampling Bituminous Materials (A.S.T.M. Designation: D 140).[1]

(*b*) *Softening Point.*—Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (A.S.T.M. Designation: D 36).[2]

(*c*) *Penetration.*—Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).[3]

(*d*) *Ductility.*—Standard Method of Test for Ductility of Bituminous Materials (A.S.T.M. Designation: D 113).[4]

(*e*) *Loss on Heating.*—Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (A.S.T.M. Designation: D 6).[5]

(*f*) *Bitumen Soluble in Carbon Disulfide.*—Standard Method of Test for the Determination of Bitumen (A.S.T.M. Designation: D 4).[6]

(*g*) *Bitumen Soluble in Carbon Tetrachloride.*—Standard Method of Test for the Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride (A.S.T.M. Designation: D 165).[7]

(*h*) *Ash.*—Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271).[8]

(*i*) *Coarse Particles.*—Tentative Method of Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (A.S.T.M. Designation: D 313 - 37 T).[9]

(*j*) *Flash Point.*—Standard Method of Test for Flash Point by Means of Open Cup (A.S.T.M. Designation: D 92).[10]

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1088.

[2] *Ibid.*, p. 1098.

[3] *Ibid.*, p. 1080.

[4] *Ibid.*, p. 1058.

[5] *Ibid.*, p. 1071.

[6] *Ibid.*, p 1041.

[7] *Ibid.*, p. 1044.

[8] 1937 Supplement to Book of A.S.T.M. Standards, p. 153.

[9] See p. 953.

[10] 1936 Book of A.S.T.M. Standards, Part II, p. 892,

TENTATIVE SPECIFICATIONS
FOR
ASPHALT FOR USE IN CONSTRUCTING BUILT-UP ROOF COVERINGS[1]

A.S.T.M. Designation: D 312 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1929; REVISED, 1935, 1937.

Scope

1. These specifications cover asphalt intended for use as hot-cement and mopping coat in the construction of built-up roof coverings for roofs surfaced in various manners, laid either over boards or concrete on various inclines, in connection with the Standard Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (A.S.T.M. Designation: D 226)[2] or the Standard Specifications for Asphalt-Saturated Asbestos Felt for Use in Constructing Built-Up Roofs (A.S.T.M. Designation: D 250)[3] of the American Society for Testing Materials.

Types

2. The asphalts covered by these specifications are as follows:

(*a*) Relatively susceptible asphalts with good adhesive and "self-healing" properties for use in slag or gravel surfaced roofing on inclines up to 3 in. per ft.;

(*b*) Somewhat less susceptible asphalts for unsurfaced roofing on inclines up to 3 in. per ft., and

(*c*) Relatively non-susceptible asphalts for use on inclines between 3 and 6 in. per ft.

Properties

3. The asphalt shall be homogeneous and free from water. It shall conform to the following requirements:

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 1137.

[3] *Ibid.*, p. 1120.

	Type of Roofing					
	Mineral Surfaced Flat		Unsurfaced Flat		Unsurfaced Steep	
	Min.	Max.	Min.	Max.	Min.	Max.
For use on decks having inclines:						
Over boards, in. per ft	....	3	....	3	3	6
Over concrete, in. per ft.:						
With nailing	....	3	....	3	3	6
Without nailing	....	1.5	....	1.5	1.5	3
(*a*) Softening point (Ring-and-Ball Method)	135 F. (57 C.)	150 F. (66 C.)	135 F. (57 C.)	170 F. (77 C.)	170 F. (77 C.)	200 F. (93 C.)
(*b*) Penetration:						
0 C. (32 F.), 200 g., 60 sec	5	....	5	....	10	....
25 C. (77 F.), 100 g., 5 sec	20	50	20	40	20	40
46 C. (115 F.), 50 g., 5 sec	100	....	....	150	....	100
(*c*) Ductility at 25 C. (77 F.), (5 cm. per min.), cm	10	....	4	....	3	....
(*d*) Loss on heating at 163 C. (325 F.), 50 g., 5 hr., per cent	....	1.5	....	1.5	....	1.5
(*e*) Penetration of residue, per cent original	60	....	60	....	60	....
(*f*) Soluble in carbon disulfide:						
Filled or native asphalt, per cent	65	....	65	....	65	....
Unfilled asphalt, per cent	99	....	99	....	99	....
(*g*) Bitumen soluble in carbon tetrachloride, per cent	99	....	99	....	99	....
(*h*) Ash:						
Filled or native asphalt, per cent	7.5	35	7.5	35	7.5	35
Unfilled asphalt, per cent	....	1	....	1	....	1
(*i*) Coarse particles retained on No. 200 sieve as percentage of matter insoluble in carbon disulfide, per cent	....	12	....	12	....	12

Sampling and Methods of Testing

4. The asphalt shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Standard Methods of Sampling Bituminous Materials (A.S.T.M. Designation: D 140).[1]

(*b*) *Softening Point.*—Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (A.S.T.M. Designation: D 36).[2]

(*c*) *Penetration.*—Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).[3]

(*d*) *Loss on Heating.*—Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (A.S.T.M. Designation: D 6).[4]

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1088.
[2] *Ibid.*, p. 1098.
[3] *Ibid.*, p. 1080.
[4] *Ibid.*, p. 1071.

(*e*) *Ductility.*—Standard Method of Test for Ductility of Bituminous Materials (A.S.T.M. Designation: D 113).[1]

(*f*) *Soluble in Carbon Disulfide.*—Standard Method of Test for the Determination of Bitumen (A.S.T.M. Designation: D 4).[2]

(*g*) *Ash.*—Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271).[3]

(*h*) *Soluble in Carbon Tetrachloride.*—Standard Method of Test for the Determination of Proportion of Bitumen Soluble in Carbon Tetrachloride (A.S.T.M. Designation: D 165).[4]

(*i*) *Coarse Particles.*—Tentative Method of Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (A.S.T.M. Designation D 313 - 37 T).[5]

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1058.

[2] *Ibid.*, p. 1041.

[3] 1937 Supplement to Book of A.S.T.M. Standards, p. 153.

[4] 1936 Book of A.S.T.M. Standards, Part II, p. 1044.

[5] See p. 953

TENTATIVE SPECIFICATIONS

FOR

ASPHALT ROOFING SURFACED WITH COARSE MINERAL GRANULES[1]

A.S.T.M. Designation: D 249 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1926; ADOPTED, 1927; REISSUED AS TENTATIVE, 1933;
REVISED, 1934, 1935, 1937.

Scope

1. These specifications cover asphalt roofing in sheet form surfaced with colored mineral granules, either 36 or 32 in. in width, composed of roofing-felt saturated and coated on both sides with asphalt and surfaced on the weather side with granulated slate or equivalent mineral material (of solid or mixed colors as may be mutually agreed upon between the purchaser and seller), and on the reverse side with powdered talc or mica.

Manufacture

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with a hot asphaltic saturant, then coated on both sides with a hot asphaltic coating compounded with a fine mineral filler substantially insoluble in water, and finally surfaced on the weather side with mineral granules embedded in the hot asphaltic coating. The reverse side shall be covered with powdered talc or mica to prevent the roofing from sticking together in the package.

Character of Felt

3. The felt shall be composed of a continuous sheet of "felted" vegetable and animal fibers. Its surface shall be uniformly smooth and free from lumps of unbeaten stock and particles of hard foreign substances.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

These specifications are in effect a revision of the former Standard Specifications for Heavy Weight Asphalt Roll-Roofing and Heavy Weight Asphalt Shingles Surfaced with Mineral Granules (A.S.T.M. Designation: D 249 – 27), which standard was accordingly discontinued in 1933.

Character of Saturant and Coatings

4. The saturant and coatings shall be composed principally of asphaltic materials.

Physical Properties

5. The fabricated product shall conform to the following requirements:

(*a*) *Width.*—The roofing shall be put up in rolls and shall be either 36 or 32 in. in width, as may be specified, within a permissible variation of ±¼ in.

(*b*) *Area.*—The average area of the rolls examined shall contain sufficient roofing to cover 100 sq. ft. of roof surface (108 sq. ft. with 2 in. or no bare lapping edge, or 111 sq. ft. with 3 in. lapping edge).

	MAX., LB.	MIN., LB.
(*c*) Weight per roll, net avg... No bare edge	....	83.0
(*c*) Weight per roll, net avg... 2 or 3 in. bare edge	....	81.0
(*d*) Weight of dry felt per 108 sq. ft.	....	10.8
(*e*) Weight of saturant (soluble in CS_2) per 108 sq. ft.[a]	....	17.3
(*f*) Weight of weather side coating (soluble in CS_2) per 108 sq. ft.	20.0	12.0
(*g*) Weight of reverse side coating (soluble in CS_2) per 108 sq. ft.	5.0	2.0
(*h*) Weight of mineral matter per 108 sq. ft. passing No. 6 and retained on No. 100 sieve	40.0	25.0
(*i*) Weight of mineral matter per 108 sq. ft. passing No. 100 sieve	10.0	4.0
(*j*) Weight of any 108 sq. ft. roll of complete roofing in the shipment (coarse granule surfaced portion)	....	80.0
(*k*) Pliability at 77 F. (25 C.)	at least 8 strips out of 10 shall not crack when bent 90 deg. at a uniform speed over a rounded corner of ¾-in. radius.	
(*l*) Behavior on heating to 176 F. (80 C.) for 2 hr	not more than 1.5 per cent volatile matter loss. The granular surfacing shall not slide more than 1/16 in when suspended vertically.	

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[a] The weight of saturant per 108 sq. ft. shall be not less than 1.6 times the weight of the dry felt.

Surface Finish

6. (*a*) The surface of the weather side shall be uniform in finish and texture. The mineral granules shall be uniformly distributed over the entire surface (except as noted in Paragraph (*b*)) in a smooth layer and shall be firmly embedded in the asphalt coating.

(*b*) The coating and granules shall cover the entire weather side of the roofing, except that this side may be provided with a bare lapping edge, approximately 2 or 3 in. in width along one edge, where either the granules alone or both the granules and coating may be omitted.

(*c*) The reverse side of the sheet shall have the asphalt coating and powdered surfacing applied uniformly to the edges.

Saturation

7. The felt shall be completely and uniformly saturated, and shall show no unsaturated spots at any point upon cutting strips at random across the entire sheet and splitting them open for their full length.

Packing

9. (*a*) The roofing shall be put up in rolls of either 108 sq. ft. when made with a 2-in. or no bare-lapping edge or 111 sq. ft. when made with a 3-in. bare-lapping edge. No roll shall contain more than two pieces nor shall there be more than 3 per cent of the rolls containing two pieces in any shipment. The rolls shall be tightly wound, and securely wrapped with a substantial grade of paper completely encircling the roll, and pasted at the overlap in such a manner as to prevent shifting from position. The ends of the rolls shall be protected with cloth covers or by other suitable means. The nails and lap-cement shall be packed in the core of each roll or furnished separately if so specified by the purchaser.

(*b*) Each roll shall be plainly marked with the manufacturer's name and brand. Special packing shall be as agreed upon between the purchaser and seller.

Nails and Lap-Cement

10. (*a*) *Nails.*—The gage of the wire shall not be greater than Washburn & Moen No. 10 (0.135 in.) nor thinner than No. 12 (0.105 in.); the head shall not be less than $\frac{3}{8}$ in. in diameter, and not less than 0.025 in. in thickness; the shank shall be between $\frac{3}{4}$ and 1 in. in length, with its lower end pointed, and shall be either smooth or barbed, and if the latter, then the barbings shall not increase the diameter of the lower two-thirds more than 0.01 in. The nails shall be zinc coated with not less than $\frac{1}{2}$ oz. nor more than $1\frac{1}{4}$ oz. per sq. ft. of nail surface. Hot-galvanized, electrogalvanized, or sherardized coatings will be accepted. There shall be furnished per "square" not less than an average of 252 nails for 36-in. roofing, and not less than an average of 275 nails for 32-in. roofing.

(*b*) *Lap-Cement.*—The cement shall be composed of bituminous materials dissolved in a volatile solvent, and shall be of such a nature as to firmly bind the laps without injurious effect upon the roofing. The cement shall have a minimum flash point of 71 F. (21.5 C.) when tested by the Tag Closed-Cup Tester. There shall be furnished for each "square" of 36-in. roofing not less than $\frac{3}{4}$ pt. of cement and for 32-in. roofing not less than 1 pt. of cement. The addition of 5 to 20 per cent by weight of short-fibered asbestos shall be permitted, in which case the quantity of cement shall be increased by $\frac{1}{4}$ pt. per "square."

(*c*) *Special Fixtures.*—Special fixtures may be furnished in lieu of nails and cement, if agreed upon by the purchaser and seller.

Sampling and Testing

11. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (A.S.T.M. Designation: D 228 – 37 T) of the American Society for Testing Materials.[1]

Inspection

12. Inspection of material shall be made as mutually agreed upon by the purchaser and seller as part of the purchase contract.

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[1] See p. 957.

TENTATIVE SPECIFICATIONS
FOR
ASPHALT ROOFING SURFACED WITH FINE MINERAL GRANULES[1]

A.S.T.M. Designation: D 248 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1926; ADOPTED, 1927; REISSUED AS TENTATIVE, 1933; REVISED, 1934, 1935, 1937.

Scope

1. These specifications cover asphalt roofing in sheet form surfaced on the weather side with fine mineral granules, either 36 or 32 in. in width, composed of roofing felt saturated and coated on both sides with asphalt.

Manufacture

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with a hot asphaltic saturant then coated on both sides with a hot asphalt coating compounded with a fine mineral filler substantially insoluble in water, and finally surfaced on the weather side with fine granules of talc or other mineral material, and of a color as may be mutually agreed upon between the purchaser and seller, embedded in the hot asphaltic coating. The reverse side shall be covered with powdered talc or mica to prevent the roofing from sticking together in the package.

Character of Felt

3. The felt shall be composed of a continuous sheet of "felted" vegetable and animal fibers. Its surface shall be uniformly smooth and free from lumps of unbeaten stock and particles of hard foreign substances.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

These specifications are in effect a revision of the former Standard Specifications for Asphalt Roll-Roofing Surfaced with Granular Talc (A.S.T.M. Designation: D 248 - 27), which standard was accordingly discontinued in 1933.

Character of Saturant and Coatings

4. The saturant and coatings shall be composed principally of asphaltic materials.

Physical Properties

5. The fabricated product shall conform to the following requirements:

(*a*) *Width.*—The roofing shall be put up in rolls and shall be either 36 or 32 in. in width, as may be specified, within a permissible variation of ±¼ in.

(*b*) *Area.*—The average area of the rolls examined shall contain not less than 108 or 216 sq. ft., as specified, which shall be sufficient to cover 100 or 200 sq. ft., respectively, of roof surface.

	GRADES			
	65 LB.		55 LB.	
	MAX., LB.	MIN., LB.	MAX., LB.	MIN., LB.
(*c*) Weight per roll, net avg. per 108 sq. ft.	....	62.0	....	52.0
(*d*) Weight of dry felt per 108 sq. ft.	....	13.5	....	10.8
(*e*) Weight of saturant (soluble in CS_2) per 108 sq. ft.[a]	....	21.6	....	17.5
(*f*) Weight of weather side coating (soluble in CS_2) per 108 sq. ft.	15.0	8.0	15.0	8.0
(*g*) Weight of reverse side coating (soluble in CS_2) per 108 sq. ft.	....	2.0	....	2.0
(*h*) Weight of mineral matter per 108 sq. ft.:				
Passing No. 14 and retained on No. 100 sieve	17.0	3.0	12.0	3.0
Passing No. 100 sieve	10.0	4.0	10.0	4.0
(*i*) Weight of any 108 sq. ft. roll of complete roofing in the shipment	....	60.0	....	50.0
(*j*) Pliability at 77 F. (25 C.)	at least 8 strips out of 10 shall not crack when bent 90 deg. at a uniform speed over a rounded corner of ½-in. radius.			
(*k*) Behavior on heating to 176 F. (80 C.) for 2 hr	not more than 1.5 per cent volatile matter loss. There shall be no flowing, sagging, blistering, or absorption of the asphalt coatings.			

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[a] The weight of saturant per 108 sq. ft. shall be not less than 1.6 times the weight of the dry felt.

Surface Finish

6. The surface shall be smooth or finely veined in appearance. Deeply ribbed surfaces shall be rejected.

Distribution of Coating and Surfacing

7. The coating and surfacing shall be applied uniformly and of approximately equal thickness on both sides and up to the edges of the sheet.

Saturation

8. The felt shall be completely and uniformly saturated, and shall show no unsaturated spots at any point upon cutting strips at random across the entire sheet and splitting them open for their full length.

Freedom from Defects

9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, rents, cracks, tears, protuberances and indentations.

Pliability and Stickiness

10. The finished product shall not crack or be so sticky as to cause tearing or material damage, upon being unrolled at atmospheric temperatures above 50 F. (10 C.).

Packing

11. (*a*) The roofing shall be put up in rolls of either 108 or 216 sq. ft. No roll shall contain more than two pieces nor shall there be more than 3 per cent of the rolls containing two pieces in any shipment. The rolls shall be tightly wound and securely wrapped in a substantial grade of paper completely encircling the roll, and pasted at the overlap in such a manner as to prevent shifting from position. The ends of the rolls shall be protected with cloth covers or by other suitable means. The nails and lap-cement shall be packed in the core of each roll or furnished separately if so specified by the purchaser.

(*b*) Each roll shall be plainly marked with the manufacturer's name and brand. Special packing shall be as agreed upon between the purchaser and seller.

Nails and Lap-Cement

12. (*a*) *Nails.*—The gage of the wire shall not be greater than Washburn & Moen No. 10 (0.135 in.) nor thinner than No. 12 (0.105 in.); the head shall not be less than $\frac{3}{8}$ in. in diameter, and not less than 0.025 in. in thickness; the shank shall be between $\frac{3}{4}$ and 1 in. in length, with its lower end pointed, and shall be either smooth or barbed, and if the latter, then the barbings shall not increase the diameter of the lower two-thirds more than 0.01 in. The nails shall be zinc coated with not less than $\frac{1}{2}$ oz. nor more than $1\frac{1}{4}$ oz. per sq. ft. of nail surface. Hot-galvanized, electrogalvanized, or sherardized coatings will be accepted. There shall be furnished per "square" not less than an average of 252 nails for 36-in. roofing, and not less than an average of 275 nails for 32-in. roofing.

(*b*) *Lap-Cement.*—The cement shall be composed of bituminous materials dissolved in a volatile solvent, and shall be of such a nature as to firmly bind the laps without injurious effect upon the roofing. The cement shall have a minimum flash point of 71 F. (21.5 C.) when tested by the Tag Closed-Cup Tester. There shall be furnished for each "square" of 36-in. roofing not less than ¾ pt. of cement and for 32-in. roofing not less than 1 pt. of cement. The addition of 5 to 20 per cent by weight of short-fibered asbestos shall be permitted, in which case the quantity of cement shall be increased by ¼ pt. per "square."

(*c*) *Special Fixtures.*—Special fixtures may be furnished in lieu of nails and cement, if agreed upon by the purchaser and seller.

Sampling and Testing

13. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (A.S.T.M. Designation: D 228 – 37 T) of the American Society for Testing Materials.[1]

Inspection

14. Inspection of material shall be made as mutually agreed upon by the purchaser and seller as part of the purchase contract.

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[1] See p. 957.

TENTATIVE SPECIFICATIONS
FOR
ASPHALT ROOFING SURFACED WITH POWDERED TALC OR MICA[1]

A.S.T.M. Designation: D 224 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1927; REISSUED AS TENTATIVE, 1933; REVISED, 1934, 1935, 1937.

Scope

1. These specifications cover asphalt roofing in sheet form surfaced with finely powdered mineral matter, either 36 or 32 in. in width, composed of roofing felt saturated and coated on both sides with asphalt.

Manufacture

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with a hot asphaltic saturant then coated on both sides with a hot asphaltic coating compounded with a finely powdered mineral filler, substantially insoluble in water, and finally surfaced on both sides with powdered talc or mica to prevent the roofing from sticking together in the package.

Character of Felt

3. The felt shall be composed of a continuous sheet of "felted" vegetable and animal fibers. Its surface shall be uniformly smooth and free from lumps of unbeaten stock and particles of hard foreign substances.

Character of Saturant and Coatings

4. The saturant and coatings shall be composed principally of asphaltic materials.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

These specifications are in effect a revision of the former Standard Specifications for **Asphalt Roll-Roofing Surfaced with Powdered Talc (A.S.T.M. Designation: D 224 - 27), which standard was accordingly discontinued in 1933.**

Physical Properties

5. The fabricated product shall conform to the following requirements:

(*a*) *Width.*—The roofing shall be put up in rolls and shall be either 36 or 32 in. in width, as may be specified, within a permissible variation of ±¼ in.

(*b*) *Area.*—The average area of the rolls examined shall contain not less than 108 or 216 sq. ft., as specified, which shall be sufficient to cover 100 or 200 sq. ft., respectively, of roof surface.

	GRADES			
	65 LB.		55 LB.	
	MAX., LB.	MIN., LB.	MAX., LB.	MIN., LB.
(*c*) Weight per roll, net avg. per 108 sq. ft.	....	62.0	....	52.0
(*d*) Weight of dry felt per 108 sq. ft.	....	13.5	....	10.8
(*e*) Weight of saturant (soluble in CS_2) per 108 sq. ft.[a]	....	21.6	....	17.3
(*f*) Weight of mineral filled coating and surface mineral matter per 108 sq. ft.	....	18.0	....	18.0
(*g*) Percentage by weight of mineral matter passing a No. 100 sieve on basis of total weight of mineral fillled coating and surface mineral matter as determined in (*f*)	50.0 per cent	15.0 per cent	50.0 per cent	15.0 per cent
(*h*) Weight of any 108 sq. ft. roll of complete roofing in the shipment	....	60.0	....	50.0

(*i*) Pliability at 77 F. (25 C.)................at least 8 strips out of 10 shall not crack when bent 90 deg. at a uniform speed over a rounded corner of ½-in. radius.

(*j*) Behavior on heating to 176 F. (80 C.) for 2 hr................................not more than 1.5 per cent volatile matter loss. There shall be no flowing, sagging, blistering or absorption of the asphalt coatings.

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[a] The weight of saturant per 108 sq. ft. shall be not less than 1.6 times the weight of the dry felt.

Surface Finish

6. The surface shall be smooth or finely veined in appearance. Deeply ribbed surfaces shall be rejected.

Distribution of Coating and Surfacing

7. The coating and surfacing shall be applied uniformly and of approximately equal thickness on both sides and up to the edges of the sheet.

Saturation

8. The felt shall be completely and uniformly saturated, and shall show no unsaturated spots at any point upon cutting strips at random across the entire sheet and splitting them open for their full length.

Freedom from Defects

9. The finished product shall be free from visible external defects, such as holes, ragged or untrue edges, rents, cracks, tears, protuberances and indentations.

Pliability and Stickiness

10. The finished product shall not crack or be so sticky as to cause tearing or material damage, upon being unrolled at atmospheric temperatures above 50 F. (10 C.).

Packing

11. (*a*) The roofing shall be put up in rolls of either 108 or 216 sq. ft. No roll shall contain more than two pieces nor shall there be more than 3 per cent of the rolls containing two pieces in any shipment. The rolls shall be tightly wound and securely wrapped in a substantial grade of paper completely encircling the roll, and pasted at the overlap in such a manner as to prevent shifting from position. The ends of the rolls shall be protected with cloth covers or by other suitable means. The nails and lap-cement shall be packed in the core of each roll or furnished separately if so specified by the purchaser.

(*b*) Each roll shall be plainly marked with the manufacturer's name and brand. Special packing shall be as agreed upon between the purchaser and seller.

Nails and Lap-Cement

12. (*a*) *Nails.*—The gage of the wire shall not be greater than Washburn & Moen No. 10 (0.135 in.) nor thinner than No. 12 (0.105 in.); the head shall not be less than $\frac{3}{8}$ in. in diameter, and not less than 0.025 in. in thickness; the shank shall be between $\frac{3}{4}$ and 1 in. in length, with its lower end pointed, and shall be either smooth or barbed, and if the latter, then the barbings shall not increase the diameter of the lower two-thirds more than 0.01 in. The nails shall be zinc coated with not less than $\frac{1}{2}$ oz. nor more than $1\frac{1}{4}$ oz. per sq. ft. of nail surface. Hot-galvanized, electrogalvanized, or sherardized coatings will be accepted. There shall be furnished per "square" not less than an average of 252 nails for 36-in. roofing and not less than an average of 275 nails for 32-in. roofing.

(*b*) *Lap-Cement.*—The cement shall be composed of bituminous materials dissolved in a volatile solvent, and shall be of such a nature as to firmly bind the laps without injurious effect upon the roofing. The cement shall have a minimum flash point of 71 F. (21.5 C.) when tested by the Tag Closed-Cup Tester. There shall be furnished for each "square" of 36-in. roofing not less than $\frac{3}{4}$ pt. of cement and for 32-in. roofing not less than 1 pt. of cement. The addition of 5 to 20 per cent by weight of short-fibered asbestos shall be permitted, in which case the quantity of cement shall be increased by $\frac{1}{4}$ pt. per "square."

(*c*) *Special Fixtures.*—Special fixtures may be furnished in lieu of nails and cement, if agreed upon by the purchaser and seller.

Sampling and Testing

13. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets and Shingles (A.S.T.M. Designation: D 228 - 37 T) of the American Society for Testing Materials.[1]

Inspection

14. Inspection of material shall be made as mutually agreed upon by the purchaser and seller as part of the purchase contract.

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[1] See p. 957.

TENTATIVE SPECIFICATIONS
FOR
ASPHALT SHINGLES SURFACED WITH COARSE MINERAL GRANULES[1]

A.S.T.M. Designation: D 225 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1925; ADOPTED IN AMENDED FORM, 1927; REVISED, 1929; REISSUED AS TENTATIVE, 1933; REVISED, 1934, 1935, 1937.

Scope

1. These specifications cover asphalt roofing in shingle form surfaced with colored mineral granules, composed of roofing felt saturated and coated on both sides with asphalt and surfaced on the weather side with granulated slate or equivalent mineral material (of solid or mixed colors as may be mutually agreed upon between the purchaser and seller), and on the reverse side with powdered talc or mica. Such roofing is intended for use as a roof covering for buildings.

Manufacture

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with a hot asphaltic saturant, then coated on both sides with a hot asphaltic coating compounded with a fine mineral filler substantially insoluble in water, and finally surfaced on the weather side with mineral granules embedded in the hot asphaltic coating. The reverse side shall be covered with powdered talc or mica to prevent the roofing from sticking together in the package.

Character of Felt

3. The felt shall be composed of a continuous sheet of "felted" vegetable and animal fibers. Its surface shall be uniformly smooth and free from lumps of unbeaten stock and particles of hard foreign substances.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

These specifications are in effect a revision of the former Standard Specifications for Asphalt Roll-Roofing and Asphalt Shingles Surfaced with Mineral Granules (A.S.T.M. Designation: D 225 - 29), which standard was accordingly discontinued in 1933.

Character of Saturant and Coatings

4. The saturant and coatings shall be composed principally of asphaltic materials.

Physical Properties

5. The fabricated product shall conform to the following requirements:

(*a*) *Shape.*—The form and size of the shingles shall be as agreed upon between the purchaser and seller.

(*b*) *Area.*—Sufficient material per "square" shall be furnished to adequately waterproof 100 sq. ft. of roof surface.

	GRADES			
	HEAVY WEIGHT		REGULAR WEIGHT	
	MAX., LB.	MIN., LB.	MAX., LB.	MIN., LB.
(*c*) Weight, net avg. per 108 sq. ft.	....	102.0	....	86.0
(*d*) Weight of dry felt per 108 sq. ft.	....	15.7	....	10.8
(*e*) Weight of saturant per 108 sq. ft.[a]	....	27.5	....	18.9
(*f*) Weight of weather side coating (soluble in CS_2) per 108 sq. ft.	20.0	12.0	20.0	12.0
(*g*) Weight of reverse side coating (soluble in CS_2) per 108 sq. ft.	5.0	2.0	5.0	2.0
(*h*) Weight of mineral matter per 108 sq. ft. passing No. 6 and retained on No. 100 sieve	40.0	25.0	40.0	25.0
(*i*) Weight of mineral matter per 108 sq. ft. passing No. 100 sieve	10.0	4.0	10.0	4.0
(*j*) Weight of complete roofing per 108 sq. ft.	....	97.0	....	83.0
(*k*) Behavior on heating to 176 F. (80 C.) for 2 hr.	not more than 1.5 per cent volatile matter loss. The granular surfacing shall not slide more than $\frac{1}{16}$ in. when suspended vertically.			

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[a] The weight of saturant per 108 sq. ft. shall be not less than 1.75 times the weight of the dry felt.

Surface Finish

6. (*a*) The weather surface shall be uniform in finish and texture. The mineral granules shall be uniformly distributed over the entire surface and so firmly embedded in the asphalt coating that when rubbed vigorously with the hand, sufficient will remain to completely cover the coating.

(*b*) The reverse side shall have a substantial coating of asphalt applied uniformly to the edges and be surfaced with powdered talc or mica.

Saturation

7. The felt shall be completely and uniformly saturated, and shall show no unsaturated spots at any point upon cutting strips 2 in. in

width across the entire shingle and splitting them open for their entire length.

Freedom from Defects

8. The finished shingles shall be free from visible external defects such as holes, ragged or untrue edges, rents, cracks, indentations or lumps of coating. The shingles shall not stick together in the packages so as to cause damage upon being unpacked at atmospheric temperatures above 50 F. (10 C).

Packing

9. (*a*) The shingles shall be put up in bundles weighing not more than 135 lb., gross. They shall be packed in cardboard cartons or between wood or cardboard ends and tied with wire or metal bands so as to make a substantial package, as may be agreed upon by the purchaser and seller.

(*b*) Each package shall be plainly marked with the manufacturer's name and brand and shall contain printed directions for laying.

Sampling and Testing

10. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (A.S.T.M. Designation: D 228 - 37 T) of the American Society for Testing Materials.[1]

Inspection

11. Inspection of material shall be made as mutually agreed upon by the purchaser and seller as part of the purchase contract.

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[1] See p. 957.

TENTATIVE SPECIFICATIONS

FOR

COAL-TAR PITCH FOR ROOFING, DAMP-PROOFING AND WATERPROOFING[1]

A.S.T.M. Designation: D 450 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover coal-tar pitch suitable for use as a mopping coat in the construction of built-up roofs surfaced with slag or gravel, as a mopping coat in damp-proofing, or as a plying or mopping cement in the construction of a membrane system of waterproofing.

Primer

2. The material used as a primer shall be creosote conforming to the Tentative Specifications for Creosote for Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing (A.S.T.M. Designation: D 43 – 37 T) of the American Society for Testing Materials.[2]

Membrane Materials

3. For the construction of built-up roofs, felts conforming to the Standard Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (A.S.T.M. Designation: D 227) of the American Society for Testing Materials[3] shall be used.

4. For the construction of a membrane system of waterproofing, any or all of the following felts, fabrics, or burlap conforming to the specifications of the American Society for Testing Materials may be used alone or in various combinations:

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[1] **Under the standardization procedure of the Society, these specifications are under the jurisdiction of the** A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

These tentative specifications are in effect a revision of, and replace the former Tentative Specifications for High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level (D 42 – 36 T), for High-Carbon Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level (D 145 – 36 T), for High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Below Ground Level (D 200 – 36 T), for High-Bitumen Coal-Tar Pitch for Use in Damp-proofing and Waterproofing Above Ground Level (D 201 – 36 T), for High-Carbon Coal-Tar Pitch for Use in Constructing Built-Up Roofs Surfaced with Slag or Gravel (D 251 – 36 T), and for High-Bitumen Coal-Tar Pitch for Use in Constructing Built-Up Roofs Surfaced with Slag or Gravel (D 252 – 36 T), which specifications were accordingly discontinued in 1937.

[2] See p. 951.

[3] 1936 Book of A.S.T.M. Standards, Part II, p. 1140.

(*a*) Standard Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (A.S.T.M. Designation: D 227).[1]

(*b*) Standard Specifications for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (A.S.T.M. Designation: D 173),[2] coal-tar saturant.

(*c*) Standard Specifications for Burlap Saturated with Bituminous Substances for Use in Waterproofing (A.S.T.M. Designation: D 174),[3] coal-tar saturant.

Types of Pitch

5. The coal-tar pitches covered by these specifications are of two types, as follows:

Type A.—Coal-tar pitch for use as a mopping coat in the construction of built-up roofs surfaced with slag or gravel on inclines not exceeding 3 in. per ft. where nailing is employed, and not exceeding 1 in. per ft. where nailing is omitted.

Coal-tar pitch for use as a mopping coat in damp-proofing or as a plying cement in the construction of a membrane system of waterproofing above ground level when not exposed to temperatures exceeding 125 F. (52 C.).

NOTE.—This type of coal-tar pitch is suitable for railroad bridges, tanks, retaining walls, culverts, dams, conduits, etc.

Type B.—Coal-tar pitch for use as a mopping coat in damp-proofing or as a plying cement in the construction of a membrane system of waterproofing below ground level under uniformly moderate temperature conditions both during the process of installation and during service.

Properties

6. The coal-tar pitch shall be homogenous and shall conform to the following requirements:

	MIN.	MAX.
(*a*) Water, per cent	....	0
(*b*) Specific gravity at 25/25 C. (77/77 F.)	1.22	1.34
(*c*) Softening point (cube-in-water method):		
Type A	140 F. (60 C.)	155 F. (68 C.)
Type B	120 F. (49 C.)	140 F. (60 C.)
(*d*) Distillation test:		
Total distillate by weight 32 to 572 F. (0 to 300 C.), per cent	....	10
Residue by weight, per cent	90	....
(*e*) Specific gravity at 100/60 F. (38/15.5 C.) of total distillate to 572 F. (300 C.)	1.03	....
(*f*) Softening point (cube-in-water method) of residue from distillation to 572 F. (300 C.)	....	175 F. (79 C.)

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1140.

[2] *Ibid.*, p. 1134.

[3] *Ibid.*, p. 1131.

	MIN.	MAX.
(*g*) Ductility at 25 C. (77 F.) (5 cm. per min.), cm....	50	....
(*h*) Total bitumen soluble in carbon disulfide, per cent	72	85
(*i*) Ash, per cent....................................	....	0.5

NOTE.—The usual range for softening point of roofing pitch shall be 140 to 150 F. In hot summer weather a softening point range of 145 to 155 F. will be permitted.

Methods of Sampling and Testing

7. The coal-tar pitch shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Standard Methods of Sampling Bituminous Materials (A.S.T.M. Designation: D 140).[1]

(*b*) *Water.*—Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (A.S.T.M. Designation: D 95).[2]

(*c*) *Specific Gravity.*—Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements and Soft Tar Pitches (A.S.T.M. Designation: D 70).[3]

(*d*) *Softening Point.*—Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (A.S.T.M. Designation: D 61).[4]

(*e*) *Distillation.*—Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment (A.S.T.M. Designation: D 20).[5]

(*f*) *Ductility.*—Standard Method of Test for Ductility of Bituminous Materials (A.S.T.M. Designation: D 113).[6]

(*g*) *Total Bitumen (Soluble in Carbon Disulfide).*—Standard Method of Test for the Determination of Bitumen (A.S.T.M. Designation: D 4).[7]

(*h*) *Ash.*—Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271).[8]

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1088.
[2] *Ibid.*, p. 997.
[3] *Ibid.*, p. 1110.
[4] *Ibid.*, p. 1103.
[5] *Ibid.*, p. 1053.
[6] *Ibid.*, p. 1058.
[7] *Ibid.*, p. 1041.
[8] 1937 Supplement to Book of A.S.T.M. Standards, p. 153.

TENTATIVE SPECIFICATIONS

FOR

CREOSOTE FOR PRIMING COAT WITH COAL-TAR PITCH IN DAMP-PROOFING AND WATERPROOFING[1]

A.S.T.M. Designation: D 43 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1917; ADOPTED IN AMENDED FORM, 1925;
REVERTED TO TENTATIVE, 1936; REVISED, 1937.

Scope

1. These specifications cover creosote primer for use, when specified, with coal-tar pitch in damp-proofing and waterproofing below or above ground level, for application to concrete and masonry surfaces.

Properties

2. Creosote primer shall be a distillate of coal-gas tar or coke-oven tar and shall conform to the following requirements:

Water	not more than 1 per cent
Consistency at 5 C. (41 F.)	entirely fluid and crystal free
Specific gravity, 38/15.5 C. (100/60 F.)	not less than 1.06
Matter insoluble in benzol	not more than 0.5 per cent
Distillation, based on water-free oil:	
Up to 210 C.	not more than 1 per cent
Up to 235 C.	not more than 10 per cent
Up to 355 C.	not less than 65 per cent
Coke residue	not more than 2 per cent

Sampling

3. (*a*) From each shipment or portion thereof representing creosote of the same kind, a number of packages shall be selected at random equivalent to the cube root of the total number of packages in the lot. If the cube root as calculated proves to be a fractional number, it shall be expressed as the next higher whole number. For convenience, the following table is given, showing the number of samples to be selected for shipments of various sizes:

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

PACKAGES IN SHIPMENT	NUMBER OF PACKAGES SELECTED	PACKAGES IN SHIPMENT	NUMBER OF PACKAGES SELECTED
2 to 8	2	217 to 343	7
9 to 27	3	344 to 512	8
28 to 64	4	513 to 729	9
65 to 125	5	730 to 1000	10
126 to 216	6	1001 to 1331	11

(*b*) By means of a paddle, the contents of each package selected shall be thoroughly stirred so as to bring all portions thereof into uniform distribution. With an appropriate sampler, not less than 1 pt. of the creosote shall be immediately drawn out from the center of each package and transferred to a clean receptacle of suitable size, which in turn shall be kept tightly covered, prior and immediately after said samples are being introduced. After all the pint samples have been added, the contents of the container shall be thoroughly agitated, whereupon 1 qt. of the mixture shall immediately be transferred to a clean quart can provided with a tightly fitting screw cap, which shall be immediately fastened in place. This sample shall be considered as representative of the shipment and shall be transmitted to the laboratory for examination, bearing a suitable tag or other means of identification.

(*c*) Creosote primer in large shipments shall be sampled in accordance with the procedure described in the Standard Methods of Sampling and Testing Creosote (A.S.T.M. Designation: D 38) of the American Society for Testing Materials.[1]

Methods of Testing

4. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Water.*—Standard Method of Test for Water in Creosote (A.S.T.M. Designation: D 370).[2]

(*b*) *Specific Gravity.*—Standard Method of Test for Specific Gravity of Creosote (A.S.T.M. Designation: D 368).[3]

(*c*) *Matter Insoluble in Benzol.*—Standard Methods of Test for Insoluble Matter in Creosote (A.S.T.M. Designation: D 367).[4]

(*d*) *Distillation.*—Standard Method of Test for Distillation of Creosote (A.S.T.M. Designation: D 246).[5]

(*e*) *Coke Residue.*—Standard Method of Test for Coke Residue of Creosote (A.S.T.M. Designation: D 168).[6]

(*f*) *Volume and Specific Gravity Correction.*—Standard Volume and Specific Gravity Correction Tables for Creosote, Creosote-Coal Tar Solution (up to 50 per cent Tar) and Coal Tar (Coke-Oven Tars) (A.S.T.M. Designation: D 347).[7]

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 531.
[2] *Ibid.*, p. 538.
[3] *Ibid.*, p. 548
[4] *Ibid.*, p. 535.
[5] *Ibid.*, p. 542.
[6] *Ibid.*, p. 553.
[7] *Ibid.*, p. 556.

TENTATIVE METHOD OF TEST
FOR
COARSE PARTICLES IN MIXTURES OF ASPHALT AND MINERAL MATTER[1]

A.S.T.M. Designation: D 313 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1929; REVISED, 1935, 1937.

Scope

1. This method covers the determination of the amount of particles of mineral or other insoluble matter in asphaltic mixtures that are retained upon a No. 200 (74-micron) A.S.T.M. sieve.

Sampling

2. The material shall be sampled in accordance with the Standard Methods of Sampling Bituminous Materials (A.S.T.M. Designation: D 140) of the American Society for Testing Materials.[2]

Preparation of Sample

3. The sample as received shall be completely melted in an oven at the lowest possible temperature, stirred well to insure homogeneity, and samples for test taken immediately.

Apparatus

4. (*a*) The apparatus shall consist either of a 3-in. No. 200 (74-micron) sieve or, a crucible sieve, $1\frac{3}{4}$ in. in diameter at the top and $1\frac{1}{4}$ in. in diameter at the bottom containing a No. 200 (74-micron) sieve. The sieve shall conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3]

(*b*) A 3-in. No. 200 (74-micron) sieve for comparison purposes shall be retained in the laboratory as a standard. Whenever a new sieve is secured, a practical test of its accuracy should be made by running on it, and on the standard sieve, a comparison test, using powdered mineral matter that has a considerable percentage of coarse particles. A reserve stock of such powdered mineral filler should be kept for this purpose.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 1088.

[3] *Ibid.*, p. 1413.

Procedure

5. (*a*) The sieve shall be dried in an oven at 105 to 110 C., cooled, and then weighed on an analytical balance, the weight being recorded to the nearest 1 mg.

(*b*) A sample of the asphalt sufficient to yield about 1 g. of mineral matter shall be weighed into a 400-ml. beaker. Then carbon disulfide, carbon tetrachloride or benzol shall be added and the mixture warmed until the asphalt is entirely dissolved. The solution obtained shall be poured through the weighed sieve, and the residue in the beaker shall be washed onto the sieve, using the solvent employed.

(*c*) The sieve containing the residue shall be washed with a fine jet of the solvent from a wash bottle until free from asphalt and fine mineral matter, then dried, tapped lightly to remove any fine mineral matter held on the wires, and weighed. During the washing, the sieve shall be held so that the liquid will drain through readily.

Percentage of Coarse Particles

6. The percentage of coarse particles in the mineral matter shall be calculated from the weight of the sample taken, the percentage of matter insoluble in carbon disulfide and the weight of the residue retained on the sieve.

TENTATIVE METHOD OF TESTING FILMS DEPOSITED FROM BITUMINOUS EMULSIONS[1]

A.S.T.M. Designation: D 466 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.[2]

RESISTANCE TO FLOW UNDER HEAT (SLIDE TEST)

Scope

1. The slide test is applicable to bituminous emulsions or reinforced emulsions intended to be applied in films of the approximate thickness described in the method and on substantially vertical surfaces.

Apparatus

2. The following apparatus will be required:

(*a*) *Brass Mask.*—A brass mask approximately $\frac{7}{64}$ in. in thickness with a rectangular opening of at least 12 sq. in.

(*b*) *Tiles.*—Unglazed ceramic tiles[3] approximately 6 by 6 in. or of sufficient size to accommodate the opening of the mask.

Procedure

3. (*a*) *Preparation of Tile.*—The tile shall be immersed in distilled water for at least 10 min. and the excess water removed immediately before application of the emulsion.

(*b*) *Preparation of Film.*—The sample of emulsion shall be thoroughly stirred. The brass mask shall then be applied to the smooth face of the tile and the emulsion spread over the area of the tile within the mask opening. The excess emulsion shall be doctored off with a flat scraper so that the film after drying averages 0.6 to 1.0 g. per sq. in. (0.10 to 0.15 g. per sq. cm.) and varies between 0.04 and 0.06 in. (1 to 1.5 mm.) in thickness.

(*c*) *Drying.*—The coated tile shall then be dried to constant weight in an atmosphere of low relative humidity at room temperature and weighed every 24 hr. A film shall be considered dry when the loss in two successive 24-hr. weighings is not greater than 0.1 g.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Accepted for publication as tentative by Committee E-10 on Standards, August 26, 1937.

[3] Suitable unglazed tile can be obtained from the Robertson Art Tile Co., Morrisville, Pa.

(*d*) *Testing.*—After drying and removing the brass mask, the coated tile shall be suspended vertically in the center of an air oven maintained at 176 F. ±5 F. (80 C. ±3 C.). The internal dimensions of the oven shall not be less than 12 by 12 by 12 in. An electrically heated oven is recommended. A thermometer shall be inserted in the oven to such depth that its bulb will be in line with the center of the specimen. The tile shall be maintained at the prescribed temperature for exactly 2 hr. Any sliding shall be determined by a reference line placed originally on the tile, coincident with the lower edge of the dried film.

RESISTANCE TO WATER ACTION

Scope

4. The purpose of this test is to determine the ability of the dried film to retain its adhesion and to resist re-emulsification after immersion in water.

Procedure

5. (*a*) *Preparation of Film.*—The film to be tested shall be prepared as described in Sections 2 and 3.

(*b*) *Testing.*—After drying, a ring approximately 2 in. in diameter and 1 in. in height shall be pressed into the surface of the coated tile. The ring shall be filled with tap water and the test specimen maintained at room temperature between 70 to 90 F. (21 to 32 C.). After 24 hr. the character of the bituminous film shall be noted by cutting with a knife or teasing needle. Adhesion or bond shall be determined by making intersecting cuts with the knife or needle and lifting the cut film at the point of intersection. Re-emulsification is indicated if the water becomes darkened by rubbing the wet surface of the film lightly with a rubber policeman.

TENTATIVE METHODS OF TESTING ASPHALT ROLL ROOFING, CAP SHEETS, AND SHINGLES[1]

A.S.T.M. Designation: D 228 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1925; ADOPTED, 1927; REISSUED AS TENTATIVE, 1930;
REVISED, 1931, 1933, 1937.

Scope

1. These methods cover procedures for the physical testing and chemical examination of roofing and shingles composed of asphalt-saturated roofing felt coated to various extents with asphalt and the coated portion surfaced with mineral powders or granules.

Types of Roofing and Shingles

2. Asphalt roll roofings, cap sheets and shingles may be divided into three types (see Fig. 1), as follows:

Type X.—A single thickness of asphalt-saturated felt, coated with asphalt compounded with a finely powdered mineral filler and surfaced with powdered mineral matter such as talc or mica.

NOTE.—Type X is commercially known as "smooth-surfaced roll roofing" and is covered by the Tentative Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica (A.S.T.M. Designation: D 224 - 37 T) of the American Society for Testing Materials.[2]

Type Y.—Similar to Type X but with the asphalt coating on the weather side surfaced with either fine granules or coarse granules.

NOTE.—When coated with fine granules or other mineral material, Type Y is commercially known as "roll roofing surfaced with granular talc" and is covered by the Tentative Specifications for Asphalt Roofing Surfaced with Fine Mineral Granules (A.S.T.M. Designation: D 248 - 37 T) of the American Society for Testing Materials.[3] When coated with coarse mineral granules this type is commercially known as "mineral-surfaced roll roofing" and is covered by the Tentative Specifications for Asphalt Roofing Surfaced with Coarse Mineral Granules (A.S.T.M. Designation: D 249 - 37 T);[4] or when cut into slabs or shingles it is commercially known as "asphalt shingles" and is covered by the Tentative Specifications for Asphalt Shingles Surfaced with Coarse Mineral Granules (A.S.T.M. Designation: D 225 - 37 T)[5] of the American Society for Testing Materials. The reverse side usually is surfaced with powdered talc or mica.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] See p. 941.

[3] See p. 937.

[4] See p. 933.

[5] See p. 945.

Type Z.—A single thickness of asphalt-saturated roofing felt, coated on the weather side for approximately one half of its width with asphalt compounded with fine mineral matter, and the coated portion surfaced with coarse mineral granules.

NOTE.—Type Z is commercially known as "mineral-surfaced cap sheet" and is covered by the Tentative Specifications for Asphalt Cap Sheet Surfaced with Coarse Mineral Granules (A.S.T.M. Designation: D 371 – 37 T) of the American Society for Testing Materials.[1]

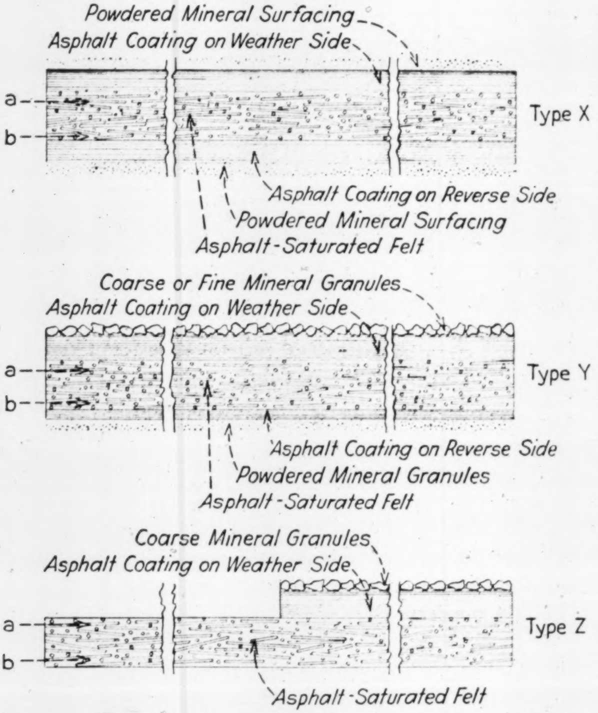
Powdered Mineral Surfacing
Asphalt Coating on Weather Side
a
b
Type X
Asphalt Coating on Reverse Side
Powdered Mineral Surfacing
Asphalt-Saturated Felt
Coarse or Fine Mineral Granules
Asphalt Coating on Weather Side
a
b
Type Y
Asphalt Coating on Reverse Side
Powdered Mineral Granules
Asphalt-Saturated Felt
Coarse Mineral Granules
Asphalt Coating on Weather Side
a
b
Type Z
Asphalt-Saturated Felt

FIG. 1.—Construction of Asphalt Roll-Roofing, Shingles, and Cap Sheets.

Sampling

3. From each shipment or portion thereof representing a product of the same kind, class, and weight, a number of rolls or bundles shall be selected at random, equivalent to one-half the cube root of the total number of rolls or bundles included in the lot, except that in lots of 1000 or less, five rolls or bundles shall be taken. If the cube root, as calculated, proves to be a fractional number, it shall be expressed as the nearest higher whole number. For convenience, the following table is given, showing the number of rolls or bundles to be selected from shipments of various sizes:

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[1] See p. 924.

PACKAGES IN SHIPMENT	NUMBER OF PACKAGES SELECTED	PACKAGES IN SHIPMENT	NUMBER OF PACKAGES SELECTED
Up to 1000	5	5 833 to 8 000	10
1001 to 1728	6	8 001 to 10 648	11
1729 to 2744	7	10 649 to 13 824	12
2745 to 4096	8	13 825 to 17 576	13
4097 to 5832	9	17 577 to 21 952	14

METHODS OF PHYSICAL TESTING

SIZE AND WEIGHT

Asphalt Roll Roofing and Cap Sheets

Packing Material, Nails, and Cement

4. Each roll selected shall be weighed to the nearest ¼ lb. The wrappers, packing material, nails, and cement from each roll shall be weighed together to the nearest ¼ lb. and the average weight thereof per "square" shall be recorded. The quantity and character of nails and cement shall be recorded.

Weight of Loose Surfacing

5. Each roll selected shall be unwound and the workmanship and finish of the roofing observed. The weight of any loose surfacing that may fall off during this operation shall be recorded.

Width, Length, Area

6. The width and length of each roll selected shall be measured to the nearest ¼ in., and the area of material contained in each roll shall be calculated in square feet. The average area of the rolls examined shall be calculated from these values.

Minimum Weight per 108 sq. ft.

7. From the information obtained under Sections 5 and 6, the net weight, exclusive of loose surfacing, of roofing per 108 sq. ft. contained in each roll selected shall be calculated. The minimum weight per 108 sq. ft. of the rolls examined shall be recorded. This shall be regarded as the minimum weight per 108 sq. ft. of the lot.

Average Weight per 108 sq. ft.

8. The average weight per 108 sq. ft. for the rolls examined shall be calculated. This shall be regarded as the average weight per 108 sq. ft. of the lot.

Selecting Average Weight Rolls

9. From the rolls examined, the one whose weight per 108 sq. ft. is nearest the average weight of the lot shall be selected. The first convolution or two shall be unrolled, and with a knife and straight edge, the sheet shall be cut across at right angles to the edges. A sample measuring exactly 30 in. in the direction of the roll's length shall then be removed. The

width of this sample shall be measured to the nearest $\frac{1}{32}$ in., and the weight of the sample in ounces determined, neglecting any loose surfacing. The weight in pounds per 108 sq. ft. shall then be calculated from the following formula:

$$\text{Weight, lb. per 108 sq. ft.} = \frac{\text{Weight of 30-in. sample in ounces}}{\text{Width of 30-in. sample in inches}} \times 32.4$$

The weight so determined shall check within 1.5 per cent, the average weight per 108 sq. ft. of the lot (Section 8). If this sample fails to check, then additional samples shall be cut from the same roll, until one is obtained which does and this sample shall be reserved for further examination as described in Sections 16 to 25.

NOTE.—As a referee method, or in case any dispute arises regarding the properties as may be ascertained from the particular sample tested, a 30-in. sample shall be taken from each roll selected in accordance with Section 3 and examined separately.

Asphalt Shingles

Weight of Packing Material

10. Each bundle selected shall be weighed to the nearest $\frac{1}{4}$ lb. The packing materials shall be weighed to the nearest $\frac{1}{4}$ lb. and the weight recorded separately for each bundle. The average weight of packing material per "square" shall be recorded.

Weight of Loose Surfacing

11. The shingles in each bundle, a, shall be counted and the workmanship and finish of the shingles shall be observed. The weight of any loose surfacing that may fall off during this operation shall be recorded.

Net Weight and Area

12. From the information obtained under Sections 10 and 11, the net weight, b, of each bundle selected, exclusive of loose surfacing, shall be calculated. The dimensions of two representative shingles shall be accurately measured and the net area, c, of material in each shingle calculated in square feet.

Minimum Weight per 108 sq. ft.

13. The area in square feet of material contained in each bundle selected shall be calculated ($a \times c$). The net weight of roofing material per 108 sq. ft. contained in each bundle shall be calculated $\left(\frac{b}{a \times c} \times 108\right)$, and the minimum weight per 108 sq. ft. recorded. This shall be regarded as the minimum weight per 108 sq. ft. of the lot.

Average Weight per 108 sq. ft.

14. The average weight, d, per 108 sq. ft. for the bundles examined shall be calculated. This shall be regarded as the average weight per 108 sq. ft. of the entire shipment.

The average weight per shingle shall be calculated $\left(\frac{c \times d}{108}\right)$.

Selecting Average Weight Shingles

15. From the bundles selected, a definite number of representative shingles whose weight per 108 sq. ft. shall fall within 1.5 per cent of the average weight of the lot (Section 14) shall be reserved for further examination in accordance with Sections 17 to 25. The number of shingles so selected shall represent as closely as possible 6 sq. ft. of material, and shall be selected as far as possible from different bundles.

NOTE.—As a referee method, or in case any dispute arises regarding the properties as may be ascertained from the particular sample tested, then a similar sample shall be taken from each bundle of shingles selected in accordance with Section 3 and examined separately.

PLIABILITY

Asphalt Roll Roofing and Cap Sheets

16. From the sample selected in accordance with Section 9, ten strips, 1 in. in width by 8 in. in length, shall be cut, five in the direction of and five across the fiber grain. They shall be immersed in water at 77 F. (25 C.) for 10 to 15 min., then removed and each strip immediately bent, with the weather side up, 90 deg. over the rounded edge of a block, at a uniform speed in approximately two seconds time. The block shall be 3 in. square by 2 in. thick with rounded corners of $\frac{1}{2}$-in. radius for Type *X* roofing and $\frac{3}{4}$ in. for Types *Y* and *Z* roofing. In bending, the strips shall be held by hand tightly against the upper 2-in. face of the block, and the projecting end of the strip shall be bent over the rounded corner without exerting any strain other than that required to keep the strip in contact with the block and to avoid kinking. Any surface ruptures exceeding $\frac{1}{8}$ in. in length shall be considered failures.

BEHAVIOR ON HEATING

Asphalt Roll Roofing, Cap Sheets and Shingles

Behavior on Heating

17. Two strips, each approximately 4 by 4 in., shall be cut from the sample selected in accordance with Section 9 in the case of asphalt roll roofing and cap sheets, and from that selected in accordance with Section 15 in the case of shingles. The strips shall be weighed and then suspended vertically, in the same direction as the material would be applied to the roof, in the center of an air oven maintained at 176 F. ± 5 F. (80 C. ± 3 C.) by means of a thin wire fastened through holes punctured near one edge. The internal dimensions of the oven shall not be less than 12 by 12 by 12 in. An electrically-heated oven is recommended. The thermometer shall be inserted in the oven to such a depth that its bulb will be in line with the

center of the specimens. The strips shall be maintained at the prescribed temperature for exactly 2 hr., then cooled in a desiccator, and each specimen weighed. The average loss of volatile matter shall be calculated as a percentage. Any change in appearance of the specimen shall be recorded, such as flowing, sagging, blistering, or absorption of the asphalt coatings; also sliding of granular surfacing.

METHODS OF CHEMICAL ANALYSIS

SEPARATION OF ROOFING OR SHINGLES INTO COMPONENT PARTS

Analysis of Roofing Composition

18. The composition of the roofing shall be analyzed as follows:

(*a*) Weight of dry felt per 108 sq. ft.,

(*b*) Weight of saturant (soluble in carbon disulfide) per 108 sq. ft.,

(*c*) Weight of weather side coating (soluble in carbon disulfide) per 108 sq. ft. (which may be either side on Type X),

(*d*) Weight of reverse side coating (soluble in carbon disulfide) per 108 sq. ft. (which may be either side on Type X),

(*e*) Weight of mineral matter per 108 sq. ft. passing a No. 6 sieve and retained on a No. 100 sieve

(*f*) Weight of mineral matter per 108 sq. ft. passing a No. 14 sieve and retained on a No. 100 sieve, and

(*g*) Weight of mineral matter per 108 sq. ft. passing a No. 100 sieve.

Preparation of Specimens for Analysis

19. (*a*) From the sample representing the average weight of the lot of roofing, as obtained in Section 9 or 15, specimens shall be cut, each measuring approximately 2 in. in width by 8 in. in length. These specimens shall be weighed, the length and width measured to within $\frac{1}{32}$ in., and the weight calculated per 108 sq. ft. Any of the 2 by 8-in. specimens whose weight varies more than 1.5 per cent from the average weight of the lot as determined in Section 8 or 14 shall be rejected. This process shall be continued until eight acceptable specimens are obtained which shall be used for determining the composition of the roofing as described in Sections 20 to 25.

(*b*) Two of the weighed 2-in. by 8-in. specimens shall each be separated into three horizontal sections approximately at the points indicated by the arrows *a* and *b* in Fig. 1. (These strips may be cut to smaller size and into disks if so desired.) The asphalt coatings with attached mineral surfacings shall be removed in such a manner that some of the saturated felt adheres to them leaving a central section of saturated felt free from coating. Any mineral surfacing detached during this operation shall be added to the section to which it belongs.

Saturant in Dry Felt

20. The saturated central horizontal sections from the two test specimens (Section 19 (*b*)) shall be weighed and then extracted with carbon disulfide in a suitable extractor or centrifuge until the washings are colorless. (Benzol having a boiling point of 80 to 82 C., carbon tetrachloride, or chloroform may be used if desired, but in case of dispute carbon disulfide shall be used.) The desaturated felt shall be dried in air, then placed in a tared weighing bottle and further dried at 105 to 110 C. (221 to 230 F.) foı 30 min., and weighed. From this weight and the original weight of the saturated felt specimens, the percentage of asphalt saturant in the dry felt shall be calculated. This figure shall be used later for calculating the weight of saturant (soluble in carbon disulfide) per 108 sq. ft. The dry felt shall be preserved for the determination of ash (Section 23).

NOTE.—While not required for testing by existing specifications, the saturant may be recovered by the method described in the paper by Gene Abson, "Method and Apparatus for the Recovery of Asphalt."[1]

Weight of Weather Side Coating Soluble in Carbon Disulfide

21. (*a*) The horizontal sections of the weather side (which may be either side on Type X roofing) containing the asphalt coating and mineral surfacing (Section 19 (*b*)) shall now be extracted with carbon disulfide in a suitable extractor until the washings are colorless. The insoluble material shall be removed and dried in air and the pieces of felt picked out and brushed free of adhering mineral matter. This felt shall then be placed in a tared weighing bottle, further dried at 105 to 110 C. (221 to 230 F.) for 30 min., and weighed. From this weight corrected for excess ash as in Section 23 and the percentage of asphalt saturant determined in Section 20, calculate the weight of asphalt in this felt. The mineral matter shall be dried in air until free from carbon disulfide, then placed in a tared weighing bottle, further dried at 105 to 110 C. (221 to 230 F.) for 30 min., cooled in a desiccator, and weighed. This mineral matter shall be retained for sieve analysis (Section 25).

(*b*) *Calculation.*—From the original weight of the sample subtract the combined weights of felt, asphalt, saturant in the felt, and mineral matter. The difference is the weight of weather side coating (soluble in carbon disulfide) per 108 sq. ft.

Weight of Reverse Side Coating Soluble in Carbon Disulfide

22. The horizontal sections of the reverse side (which on Type X roofing will be the side opposite to that selected under Section 21) containing the coating and mineral surfacing (Section 19 (*b*)) shall now be extracted with carbon disulfide and the weight of coating soluble in carbon disulfide calculated as in Section 21. The mineral matter recovered shall be combined

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 704 (1933).

with that obtained from the weather side (Section 21) and retained for sieve analysis (Section 25). The felt shall be ashed as in Section 23, in order to determine the true weight of felt.

Ash of Desaturated Felt

23. The desaturated felt obtained in Sections 20, 21 and 22 from the center, and weather and reverse sides shall be ashed separately in weighed crucibles either over an open flame or in a muffle furnace until all carbon has been consumed. The weight shall be determined. The percentage of ash in the center portion (Section 20) will be assumed to be the true percentage of ash of the entire felt. The excess ash in the weather side portion (Section 21) will be added to the mineral matter passing a No. 100 sieve as determined in Section 25. This weight will also be used for correcting the total weight of desaturated felt and weight of saturant soluble in carbon bisulfide. In like manner, the excess ash in the reverse side portion will be added to the mineral matter passing a No. 100 sieve and for correcting the total weights of felt and saturant soluble in carbon disulfide.

Weight of Dry Desaturated Felt per 108 sq. ft.

24. The combined weights of the felt from the center portion obtained in Section 20 and the weights of the felts from the weather side and reverse side portions obtained in Sections 21 and 22, respectively, corrected for excess ash (Section 23) shall be the total weight of dry felt in the two 2-in. by 8-in. specimens. From this weight calculate the weight of dry felt per 108 sq. ft.

Weight of Mineral Matter

25. The total mineral matter recovered in Sections 21 and 22 plus the excess ash (Section 23) shall be moistened with a few drops of alcohol, boiled with 100 ml. of water and washed successively through No. 6, No. 14, and No. 100 sieves with sufficient hot water to remove the fine mineral matter. The mineral matter retained on each sieve shall be collected separately, dried at 105 to 110 C. (221 to 230 F.) for 30 min., and weighed. From these weights, the weight of mineral matter shall be calculated and reported as follows:

Weights of Mineral Matter per 108 sq. ft.:
Passing a No. 6 sieve, retained on a No. 100 sieve............ ....
Passing a No. 14 sieve, retained on a No. 100 sieve........... ....
Passing a No. 100 sieve (by difference)...................... ....

To check the weight of mineral matter passing a No. 100 sieve, the total aqueous suspension of mineral matter which passes through the No. 100 sieve shall be recovered by filtering through a weighed Gooch crucible, dried at 105 to 110 C. (221 to 230 F.), and weighed.

TENTATIVE METHOD OF TEST
FOR
SIEVE ANALYSIS OF GRANULAR MINERAL SURFACING FOR ASPHALT ROOFING AND SHINGLES[1]

A.S.T.M. Designation: D 451 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. This method is intended for the sieve analysis of granular mineral surfacing material, such as crushed slate, stone, etc., used on the weather surface of prepared asphalt roofing and shingles.

Sieves

2. (*a*) A set of consecutive sieves of the series listed in Table I, conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials,[2] shall be used. The group of sieves selected shall include those appropriate to the grading of the granular mineral to be subjected to sieve analysis; sieves of either the coarser or finer openings, on which less than

TABLE I.—REQUIREMENTS FOR SIEVE OPENINGS AND WIRE DIAMETERS WITH PERMISSIBLE VARIATIONS.

Sieve Designation	Corresponding U. S. Standard Sieve Series Number	Sieve Opening		Wire Diameter		Tolerance in Average Opening, per cent	Tolerance on Wire Diameter, per cent		Tolerance in Maximum Opening, per cent
		mm.	in.	mm.	in.		Under	Over	
3360 micron	6	3.36	0.132	1.02	0.040	±3	15	30	10
2380 micron	8	2.38	0.0937	0.84	0.0331	±3	15	30	10
1680 micron	12	1.68	0.0661	0.69	0.0272	±3	15	30	10
1190 micron	16	1.19	0.0469	0.54	0.0213	±3	15	30	10
840 micron	20	0.84	0.0331	0.42	0.0165	±5	15	30	25
590 micron	30	0.59	0.0232	0.33	0.0130	±5	15	30	25
420 micron	40	0.42	0.0165	0.25	0.0098	±5	15	30	25
297 micron	50	0.297	0.0117	0.188	0.0074	±6	15	35	40
210 micron	70	0.210	0.0083	0.140	0.0055	±6	15	35	40
149 micron	100	0.149	0.0059	0.102	0.0040	±6	15	35	40

NOTE.—Sieves of the Tyler standard screen scale series, having mesh designations: Nos. 6, 8, 10, 14, 20, 28, 35, 48, 65 and 100, conform, respectively, to the requirements in Table I for sieve opening and wire diameter, within the tolerances stated, for the sieves of the U. S. Standard Sieve Series, in the order listed.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

0.05 per cent of the weight of the sample of the particular material would be found after sieving, need not be included in the set.

(*b*) Sieves shall be wire cloth, square-woven, from brass, bronze, or other suitable wire, mounted without distortion in circular frames, about 20 cm. (8 in.) in diameter and about 5 cm. (2 in.) between the top of the frame and the cloth.

Sieve Shaker

3. A mechanically operated sieve shaker,[1] which imparts to the set of sieves a rotary motion and tapping action of uniform speed, shall be used. The number of taps per minute shall be between 140 and 160. The sieve shaker shall be fitted with a hard maple plug to receive the impact of the tapping device. The entire apparatus shall be rigidly mounted by bolting to a solid foundation, preferably of concrete.

Sample Splitter

4. A riffle sampler with $\frac{3}{8}$ or $\frac{1}{2}$-in. divisions, for reducing the gross sample to the quantity required for the sieve analysis, shall be used.

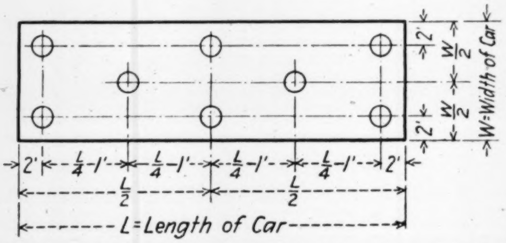

FIG. 1.—Location of Sampling Points from Surface of Car.

Unit for Sampling

5. Each carload of mineral granules shall be considered the unit for sampling, except that where a carload includes more than one kind of granular surfacing, the entire quantity of each kind in the car shall be considered the unit for each kind, respectively.

Gross Sample

6. (*a*) In collecting the gross sample from a carload of mineral granules shipped in bulk, eight equal increments of not less than 2 lb. each shall be taken from the material at the exposed surface of the car. A conical excavation about 1 ft. in depth shall be made at each sampling point and the sample taken from the bottom of the excavation. The eight sampling points shall be located as shown in Fig. 1.

(*b*) In collecting the gross sample from a carload of mineral granules shipped in bags, a number of bags shall be selected at random, equivalent to the cube root of the total number of bags in the car. The gross sample shall consist of equal increments of not less than 2 lb. from each of the bags taken for sampling.

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[1] One such sieve shaker meeting the requirements prescribed in Section 3 is the "Rotap," manufactured by the W. S. Tyler Co., Cleveland, Ohio.

(*c*) Where the mineral granules are being loaded or unloaded by conveyor or chute the gross sample shall preferably be collected by taking equal increments from the stream at regular time intervals, with such frequency that the total weight of the gross sample will be not less than 16 lb. Increments shall be taken from the full width and thickness of the stream, preferably, if practicable, by inserting a suitable container into the stream.

Reduction of Gross Sample

7. The gross sample shall be reduced by riffling or hand-quartering to not less than 500 g. (1.1 lb.).

Size of Test Sample

8. The entire sample obtained from reduction of the gross sample, shall be weighed with an accuracy of not less than 0.1 g., and taken for the sieve analysis.

Sieve Analysis

9. (*a*) The group of sieves selected from the series listed in Table I shall be assembled in consecutive order as to size of openings, with the sieve having the largest openings at the top and the one with the smallest openings at the bottom, the assembly being completed by a solid collecting pan below the bottom sieve. The test sample, previously weighed (Section 8), shall be placed on the top sieve and this sieve closed with a solid cover. The sieve assembly shall then be securely fastened in a suitable mechanical sieve shaking device (Section 3).

(*b*) The sample shall be passed through the sieves of the series selected, by subjecting it to the action of the sieve shaker for a period of 15 min. Since granular mineral surfacing materials usually have been subjected to grading by screens in the process of manufacture and hence separate rapidly into their sieve fractions, an end point determination is not considered necessary.

(*c*) The portion of the sample retained on each of the sieves and on the pan shall be carefully removed and weighed with an accuracy of not less than 0.1 g.

Report

10. The results of the sieve analysis shall be reported to the nearest 0.1 per cent (omitting the results on those sieves on which less than 0.05 per cent of the total weight of the test sample was collected), as follows:

	PER CENT
Retained on No. 6 (3360-micron) sieve..	....
Retained on No. 8 (2380-micron) sieve, passing No. 6 (3360-micron) sieve.......	....
Retained on No. 12 (1680-micron) sieve, passing No. 8 (2380-micron) sieve......	....
Retained on No. 16 (1190-micron) sieve, passing No. 12 (1680-micron) sieve.....	....
Retained on No. 20 (840-micron) sieve, passing No. 16 (1190-micron) sieve......	....
Retained on No. 30 (590-micron) sieve, passing No. 20 (840-micron) sieve.......	....
Retained on No. 40 (420-micron) sieve, passing No. 30 (590-micron) sieve.......	....
Retained on No. 50 (297-micron) sieve, passing No. 40 (420-micron) sieve.......	....
Retained on No. 70 (210-micron) sieve, passing No. 50 (297-micron) sieve.......	....
Retained on No. 100 (149-micron) sieve, passing No. 70 (210-micron) sieve......	....
Passing No. 100 (149-micron) sieve.....	....
Total..	....

The sum of the sieve weight percentage fractions, reported as above tabulated, shall be not less than 99.5 per cent, that is, the total loss and error in the sieve analysis shall not exceed 0.5 per cent.

Reproducibility of Results

11. Duplicate determinations by the same operator, using the same sieves, shall check within 1 per cent of the total weight of the sample, for the portion of the sample retained on each sieve and on the pan.

TENTATIVE METHOD OF TEST

FOR

SIEVE ANALYSIS OF NON-GRANULAR MINERAL SURFACING FOR ASPHALT ROOFING AND SHINGLES[1]

A.S.T.M. Designation: D 452 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. This method is intended for the sieve analysis of non-granular mineral surfacing material, such as mica, talc and other powdered or flaky mineral particles, used on the weather surfaces of prepared asphalt roofing and on the non-weather-exposed surface of asphalt shingles.

Sieves

2. (*a*) A set of consecutive sieves of the series listed in Table I, conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials,[2] shall be used. The group of sieves selected shall include those appropriate to the grading of the non-granular mineral to be subjected to sieve analysis; sieves of either the coarser or finer openings, on which

TABLE I.—REQUIREMENTS FOR SIEVE OPENINGS AND WIRE DIAMETERS WITH PERMISSIBLE VARIATIONS.

Sieve Designation	Corresponding U. S. Standard Sieve Series Number	Sieve Opening		Wire Diameter		Tolerance in Average Opening, per cent	Tolerance on Wire Diameter, per cent		Tolerance in Maximum Opening, per cent
		mm.	in.	mm.	in.		Under	Over	
1680 micron	12	1.68	0.0661	0.69	0.0272	±3	15	30	10
1190 micron	16	1.19	0.0469	0.54	0.0213	±3	15	30	10
840 micron	20	0.84	0.0331	0.42	0.0165	±5	15	30	25
590 micron	30	0.59	0.0232	0.33	0.0130	±5	15	30	25
420 micron	40	0.42	0.0165	0.25	0.0098	±5	15	30	25
297 micron	50	0.297	0.0117	0.188	0.0074	±6	15	35	40
210 micron	70	0.210	0.0083	0.140	0.0055	±6	15	35	40
149 micron	100	0.149	0.0059	0.102	0.0040	±6	15	35	40
74 micron	200	0.074	0.0029	0.053	0.0021	±8	15	35	60

NOTE.—Sieves of the Tyler standard screen scale series, having mesh designations: Nos. 10, 14, 20, 28, 35, 48, 65, 100 and 200, conform, respectively, to the requirements in Table I for sieve opening and wire diameter, within the tolerances stated, for the sieves of the U. S. Standard Sieve Series, in the order listed.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 1413.

less than 0.05 per cent of the weight of the sample of the particular material would be found after sieving, need not be included in the set.

NOTE.—For relatively coarse or flaky materials, such as coarse mica, sieves from No. 16 to No. 70, inclusive, of the U. S. Standard series will usually be found suitable; for finer surfacing materials, such as fine mica or talc flour, sieves from No. 30 to No. 200, inclusive, of the U. S. Standard series will usually give a satisfactory sieve analysis.

(*b*) Sieves shall be wire cloth, square-woven, from brass, bronze, or other suitable wire, mounted without distortion in circular frames, about 20 cm. (8 in.) in diameter and about 5 cm. (2 in.) between the top of the frame and the cloth.

Sieve Shaker

3. A mechanically operated sieve shaker,[1] which imparts to the set of sieves a rotary motion and tapping action of uniform speed, shall be used. The number of taps per minute shall be between 140 and 160. The sieve shaker shall be fitted with a hard maple plug to receive the impact of the tapping device. The entire apparatus shall be rigidly mounted by bolting to a solid foundation, preferably of concrete.

Sample Splitter

4. A riffle sampler with $\frac{3}{8}$ or $\frac{1}{2}$-in. divisions, for reducing the gross sample to the quantity required for the sieve analysis, shall be used.

Unit for Sampling

5. Each carload of non-granular mineral surfacing shall be considered the unit for sampling.

Gross Sample

6. (*a*) In collecting the gross sample from a carload of non-granular mineral surfacing shipped in bags, a number of bags shall be selected at random equivalent to the cube root of the total number of bags in the car. The gross sample shall consist of equal increments of not less than 0.5 lb. from each of the bags taken for sampling.

(*b*) In collecting the gross sample from a carload of non-granular mineral surfacing material shipped in bulk, at least eight equal increments of not less than 0.5 lb. each shall be taken from well distributed points in the mass, using a sampling tube not less than 1 in. in diameter. A conical excavation about 1 ft. in depth shall be made at each sampling point and the sampling tube inserted into the mass at the bottom of the excavation.

Reduction of Gross Sample

7. The gross sample shall be reduced by riffling or hand-quartering to not less than 100 g. (0.22 lb.).

Size of Test Sample

8. The entire sample obtained from reduction of the gross sample,

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[1] One such sieve shaker meeting the requirements prescribed in Section 3 is the "Rotap," manufactured by the W. S. Tyler Co., Cleveland, Ohio.

shall be dried at least 2 hr. in an oven at 220 F., weighed with an accuracy of not less than 0.1 g., and taken for the sieve analysis.

Sieve Analysis

9. (*a*) The group of sieves selected from the series listed in Table I shall be assembled in consecutive order as to size of openings, with the sieve having the largest openings at the top and the one with the smallest openings at the bottom, the assembly being completed by a solid collecting pan below the bottom sieve. The test sample, previously weighed (Section 8), shall be placed on the top sieve and this sieve closed with a solid cover. The sieve assembly shall then be securely fastened in a suitable mechanical sieve shaking device (Section 3).

(*b*) The sample shall be passed through the sieves of the series selected, by subjecting it to the action of the sieve shaker for a period of 20 min. At the end of this period the collecting pan, containing the portion of the material passing the finest sieve of the group selected, shall be removed from the sieve assembly and the contents weighed with an accuracy of not less than 0.1 g. The collecting pan shall then be re-assembled with the sieves, as before, and the shaking continued for an additional 10 min. At the end of this additional shaking period the collecting pan shall be removed and the contents weighed. If the additional material passing the finest sieve during this second shaking period does not exceed 0.5 per cent of the total weight of the sample, the sieve analysis shall be considered complete. If it does exceed 0.5 per cent, the collecting pan and sieves shall be re-assembled and shaken for successive additional 10-min. periods, weighing the material collected in the pan after each period of shaking, until the amount passing the finest sieve in a 10-min. shaking period is less than 0.5 per cent of the weight of the sample.

(*c*) The portion of the sample retained on each of the sieves and on the pan shall be carefully removed and weighed with an accuracy of not less than 0.1 g.

Report

10. The results of the sieve analysis shall be reported to the nearest 0.1 per cent (omitting the results on those sieves on which less than 0.05 per cent of the total weight of the test sample was collected), as follows:

	PER CENT
Retained on No. 12 (1680-micron) sieve	....
Retained on No. 16 (1190-micron) sieve, passing No. 12 (1680-micron) sieve	....
Retained on No. 20 (840-micron) sieve, passing No. 16 (1190-micron) sieve	....
Retained on No. 30 (590-micron) sieve, passing No. 20 (840-micron) sieve	....
Retained on No. 40 (420-micron) sieve, passing No. 30 (590-micron) sieve	....
Retained on No. 50 (297-micron) sieve, passing No. 40 (420-micron) sieve	....
Retained on No. 70 (210-micron) sieve, passing No. 50 (297-micron) sieve	....
Retained on No. 100 (149-micron) sieve, passing No. 70 (210-micron) sieve	....
Retained on No. 200 (74-micron) sieve, passing No. 100 (149-micron) sieve	....
Passing No. 200 (74-micron) sieve	....
Total	....

The sum of the sieve weight percentage fractions, reported as above tabulated, shall not be less than 98.5 per cent, that is, the total loss and error in the sieve analysis shall not exceed 1.5 per cent.

Reproducibility of Results

11. Duplicate determinations by the same operator, using the same sieves, shall check within 1 per cent of the total weight of the sample, for the portion of the sample retained on each sieve and on the pan.

TENTATIVE SPECIFICATIONS
FOR
PHENOLIC LAMINATED SHEET FOR RADIO APPLICATIONS[1]

A.S.T.M. Designation: D 467 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.[2]

Scope

1. These specifications cover phenolic laminated sheet for radio or similar applications where high frequencies are involved. The following grades are included:

Grade No. 1.—A paper base punching stock where dielectric losses are of minor importance.
Grade No. 2.—A paper base punching stock with medium dielectric loss.
Grade No. 3.—A paper base punching stock with low dielectric loss.
Grade No. 4.—A hard paper base stock with low dielectric loss.
Grade No. 5.—A fabric base stock with medium dielectric losses.
Grade No. 6.—A combination paper and fabric base stock with one fabric sheet on one surface. This material has medium dielectric losses.

NOTE.—**Usual applications for the various grades are given in the Appendix.**

MANUFACTURE

Material

2. The material shall consist of layers of paper or cotton fabric as required. These layers shall be suitably impregnated with a phenolic type resinous compound and consolidated into hard dense sheets under the action of heat and pressure.

PHYSICAL PROPERTIES AND TESTS

Punching Quality

3. (*a*) The grades of material differ in their suitability for punching but any of the grades may be punched in simple shapes and thin sizes provided good punching practice is used, including sharp, close clearance dies, proper stripper plates, and proper heating conditions. When using good punching practice as outlined in Paragraph (*c*), the various grades shall

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

[2] Accepted for publication as tentative by Committee E-10 on Standards, August 26, 1937.

punch satisfactorily in thicknesses up to and including the maximum limits given in Table I.

(*b*) Where punching properties better than those listed in Table I are required for particular parts, this shall be subject to agreement between the manufacturer and the purchaser.

(*c*) In good punching practice, the edges of the piece shall not be closer to the edge of the strip than twice the thickness of the sheet; the holes shall not be smaller in diameter than the thickness of the sheet nor have square corners; the **distance between holes or between holes and edge of piece shall not be less** than the thickness of the sheet. Heating the material to a temperature of 120 to 140 C. (approximately 15 min. for material $\frac{1}{8}$ in. in thickness) is generally necessary for best punching results, although in Grade No. 1 this may make the material too soft. In this case, better results may be obtained by heating at lower temperatures or for a shorter time. If more than 2 min. elapse between the time the strip leaves the heating medium and the last piece is punched, results will be poor.

Table I.—Maximum Thickness for Various Grades at Which Satisfactory Punching Results May Be Obtained when Using Good Punching Practice.

Grade	Room Temperature (20 to 30 C.)	Heated to 120 to 140 C. Before Punching
No. 1	$\frac{1}{16}$ in.	$\frac{1}{8}$ in.
No. 2	$\frac{1}{32}$ in.	$\frac{3}{32}$ in.
No. 3	......	$\frac{1}{16}$ in.
No. 4	......	$\frac{1}{16}$ in. in simple shapes only
No. 5	......	$\frac{1}{8}$ in.
No. 6	......	$\frac{3}{32}$ in.

Note.—With simple forms and special precautions greater thicknesses than the above can sometimes be punched. With poor dies, poor punching practice or intricate parts, good results cannot be expected in the thicknesses listed in the table.

Flexural Strength

4. The flexural strength in either direction of the sheet, tested flatwise, shall not be less than 11,000 lb. per sq. in. The flexural strength shall be determined in accordance with the Tentative Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229 - 37 T) of the American Society for Testing Materials.[1]

Electrical Properties and Tests

Power Factor, Dielectric Constant and Dielectric Loss Factor

5. (*a*) The values for power factor, dielectric constant and dielectric loss factor when measured at a frequency of 1,000,000 cycles shall not exceed those given in Table II.

(*b*) The apparatus and general method of test shall conform to the Tentative Methods of Testing Electrical Insulating Materials for Power

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[1] See p. 1027.

Factor and Dielectric Constant (A.S.T.M. Designation: D 150 - 36 T) of the American Society for Testing Materials.[1] The sizes of specimens and electrodes shall be as listed in Table III. The test on material as received shall be made on specimens under ordinary laboratory conditions provided the temperature is between 20 and 30 C. The immersion for 24 hr. in water shall be made in a fresh supply of clean tap water held at 25 C. ± 2 C. For material $\frac{1}{4}$ in. and under in thickness duplicate specimens from the same sheet shall be used, one for the "as received" test and the other for the water-immersion test. For thicker materials, the "as received" specimen may be used for water immersion, provided the disk electrode is removed, the grease washed off with benzol or gasoline and the sample wiped carefully and allowed to air dry for 1 hr. before placing it in the water. In all cases, specimens shall be removed from the water at the end of 24 hr. and the excess water wiped off with a soft cloth; then the electrodes shall be applied and measurements made immediately.

TABLE II.—MAXIMUM PERMISSIBLE VALUES FOR POWER FACTOR, DIELECTRIC CONSTANT AND DIELECTRIC LOSS FACTOR MEASURED PERPENDICULAR TO LAMINATIONS.

Frequency = 1,000,000 cycles.

Grade	Values on Material as Received			Values on Material After 24 hr. in Water	
				Thicknesses $\frac{3}{64}$ in. and over	Thickness up to $\frac{3}{64}$ in.
	Power Factor	Dielectric Constant	Dielectric Loss Factor	Power Factor	Power Factor
No. 1	0.060	....	....	0.100	0.150
No. 2	0.045	5.5	0.25	0.050	0.060
No. 3	0.030	5.2	0.16	0.035	0.045
No. 4	0.035	5.2	0.18	0.040	......
No. 5	0.045	5.5	0.25	0.050	......
No. 6	0.045	5.5	0.25	0.050	......

TABLE III.—SIZE OF SPECIMENS AND ELECTRODES FOR MEASUREMENTS OF POWER FACTOR AND DIELECTRIC CONSTANT.

THICKNESS, IN.	DIAMETER OF DISK ELECTRODES, IN.	SIZE OF SPECIMEN, IN.
Under $\frac{1}{16}$	3	6 by 6
$\frac{1}{16}$ to $\frac{1}{8}$, inclusive	4.5	6 by 6
Over $\frac{1}{8}$ to $\frac{1}{4}$, inclusive	6	8 by 8
Over $\frac{1}{4}$ to $\frac{1}{2}$, inclusive	8	10 by 10
Over $\frac{1}{2}$ to 1, inclusive	11	14 by 14

STANDARD SIZES AND DIMENSIONS

Size of Sheet

6. Sheets shall be furnished in manufacturer's standard sheet sizes. The length and width may vary ± 1 in. from the manufacturer's standard size.

NOTE.—In general, manufacturer's standard widths are between 36 and 48 in.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 955 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1069.

Permissible Variations in Size of Small Pieces

7. When small pieces are ordered to be cut from standard size sheets the permissible variations from the specified length or width shall be within the requirements shown in Table IV.

TABLE IV.—PERMISSIBLE VARIATIONS IN SIZE OF SMALL PIECES.

NOMINAL THICKNESS, IN.	PERMISSIBLE VARIATIONS IN LENGTH OR WIDTH, IN.
$\frac{1}{64}$ to $\frac{1}{4}$, inclusive	±0.015
$\frac{17}{64}$ to $\frac{1}{2}$, inclusive	±0.017
$\frac{33}{64}$ to 1, inclusive	±0.020

Standard Thicknesses

8. The nominal thickness of the material shall be specified on the order. The standard ranges of thicknesses for each grade are shown in Table V.

TABLE V.—STANDARD RANGES OF THICKNESSES AVAILABLE.

GRADE	RANGE OF THICKNESS, IN.
No. 1	$\frac{1}{64}$ to $\frac{3}{32}$, inclusive
No. 2	$\frac{1}{32}$ to $\frac{1}{4}$, inclusive
No. 3	$\frac{1}{32}$ to $\frac{1}{4}$, inclusive
No. 4	$\frac{1}{16}$ to 1, inclusive
No. 5	$\frac{1}{16}$ to $\frac{1}{2}$, inclusive
No. 6	$\frac{3}{64}$ to $\frac{3}{32}$, inclusive

TABLE VI.—PERMISSIBLE VARIATIONS IN THICKNESS.

Nominal Thickness,[a] in.		Permissible Variations, in., Plus or Minus	
Fraction of Inch	Decimal Equivalent	Grades Nos. 1, 2, 3, 4 and 6	Grade No. 5
$\frac{1}{64}$	0.0155	0.0025	......
$\frac{1}{32}$	0.031	0.0035	......
$\frac{3}{64}$	0.047	0.0045	......
$\frac{1}{16}$	0.0625	0.0055	0.0075
$\frac{3}{32}$	0.094	0.007	0.009
$\frac{1}{8}$	0.125	0.008	0.010
$\frac{5}{32}$	0.156	0.009	0.011
$\frac{3}{16}$	0.1875	0.010	0.0125
$\frac{7}{32}$	0.219	0.011	0.014
$\frac{1}{4}$	0.250	0.012	0.015
$\frac{5}{16}$	0.3125	0.0145	0.0175
$\frac{3}{8}$	0.375	0.017	0.020
$\frac{7}{16}$	0.438	0.019	0.022
$\frac{1}{2}$	0.500	0.021	0.024
$\frac{5}{8}$	0.625	0.024	......
$\frac{3}{4}$	0.750	0.027	......
$\frac{7}{8}$	0.875	0.030	......
1	1.000	0.033	......

[a] On sheet of nominal thickness not listed in the above table, the permissible variations shall be the same as given for the next greater thickness.

Permissible Variations in Thickness

9. The permissible plus or minus variations from standard thicknesses of the various grades are given in Table VI. At least 90 per cent of the

area of the sheet shall be within the variations listed and at no point shall the thickness as measured vary from the nominal by a value greater than 125 per cent of the specified variation.

Workmanship and Finish

Workmanship

10. The material shall be uniform in quality consistent with the properties prescribed in these specifications. It shall be free from blisters, wrinkles or cracks and reasonably free from other small defects such as scratches, dents, solvent areas, etc.

Color and Finish

11. The color and finish of the material shall be as specified in the order. The standard colors and finishes for the various grades are listed in Table VII.

Table VII.—Standard Colors and Finishes Available.

Grade	Color[a]	Finish
No. 1	Natural, Black, Chocolate	Semigloss or Dull
No. 2	Natural, Black, Chocolate	Semigloss or Dull
No. 3	Natural only	Semigloss or Dull
No. 4	Natural or Black	Polished or Semigloss
No. 5	Natural only	Semigloss
No. 6	Natural on fabric surface Chocolate on paper surface	Semigloss

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[a] *Natural* is the color produced by the natural undyed paper or fabric and resin used and may vary from a light tan to a light brown or reddish brown. This natural color shall be free from streaks or stains, and substantially uniform except for Grade No. 3 which may be mottled in appearance.

Black sheets shall have substantially uniform black surfaces and black body. A slight grayish-black color on sawed or machined edges is permissible.

Chocolate.—Uniform dark brown or chocolate surface sheets with natural core.

Packing and Marking

12. Unless otherwise specified, material shall be packed in substantial crates or boxes in accordance with the usual practice of the individual manufacturer.

Marking

13. Every full size sheet shall be marked with the manufacturer's name or trade-mark, the A.S.T.M. grade number and **designation D 467 – 37 T.** The containers shall be marked with the manufacturer's name, A.S.T.M. grade number **and designation D 467 – 37 T,** size and quantity of sheets therein, together with other marking required by the purchaser.

Inspection

14. (*a*) The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with these specifications. Tests and inspection at the place of manufacture may be made prior to shipment, and such inspection shall be final. All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

(*b*) In case material is not inspected at the place of manufacture, the purchaser may make the tests to govern the acceptance or rejection of the material in his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

Rejection

15. (*a*) Unless otherwise specified, any rejections based on tests made in accordance with Section 14 (*b*) shall be reported to the manufacturer within three weeks from the receipt of samples by the purchaser.

(*b*) Any portion of an accepted shipment of material which is subsequently found not to be in accordance with these specifications, will be rejected, provided the manufacturer is notified within 90 days from the date of receipt of the material by the purchaser.

(*c*) Samples tested in accordance with Section 14 (*b*) which represent rejected material, shall be preserved for one month from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

APPENDIX

USUAL APPLICATIONS FOR THE VARIOUS GRADES OF PHENOLIC LAMINATED SHEETS

Grade No. 1.—Sockets, terminal boards, miscellaneous instrument parts where insulating properties and moisture resistance are of minor importance and good punching and assembling characteristics essential.

Grade No. 2.—General use for insulation in radio frequency circuits where punching and assembling operations are not particularly severe.

Grade No. 3.—Simple punched parts where very low losses are essential such as variable capacitor insulation and metal tube insulation and some range switch parts.

Grade No. 4.—General use in instruments and other applications where parts are produced by machining and where reasonably good appearance and good electrical properties are desired.

Grade No. 5.—Applications similar to Grade No. 2 except for more severe punching and assembling operations.

Grade No. 6.—For tube sockets and other applications similar to Grade No. 2 where slightly better punching quality and toughness are desirable and where tendency to warp is not objectionable.

TENTATIVE SPECIFICATIONS

FOR

BLACK BIAS-CUT VARNISHED CLOTH TAPE USED FOR ELECTRICAL INSULATION[1]

A.S.T.M. Designation: D 373 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1933; REVISED, 1934, 1936, 1937.

Scope

1. These specifications cover black seamless bias-cut varnished cloth tapes 7 to 12 mils in thickness to be used for insulating bus bars, joints and terminals of electric wires and cables, and other electrical apparatus connections.

NOTE.—Orders for material under these specifications shall include the following information:

The desired nominal thickness, width and length (Sections 6, 7 and 8);
Type of surface, greasy or tacky (Section 11);
Type of packing, dry or oil (Sections 17, 18 and 19);
Number of rolls in package (Sections 18 and 19); and
Marking on package (Section 20).

MANUFACTURE

Cloth and Varnish

2. The cloth and the number of coats of varnish applied shall be such as to produce a product which will meet the requirements of these specifications.

Defects

3. In general, the tape shall be free from wrinkles, excessive pebbling, blisters, and other imperfections.

Splices

4. Splices are those joints made by the manufacturer of the untreated cloth. Splices shall not occur more than once in each roll, except that rolls having a length of 6 yd. or less shall contain no splices.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

METHODS OF TESTING

Methods of Testing

5. (*a*) Varnished cloth tape shall be sampled, conditioned and tested in accordance with the Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (A.S.T.M. Designation: D 295 - 37 T) of the American Society for Testing Materials.[1]

(*b*) All specimens taken from a single roll shall be numbered or marked so that failures can be identified with the roll.

DIMENSIONS AND PERMISSIBLE VARIATIONS

Thickness

6. The nominal thickness shall be specified in the purchase order. The average thickness shall be determined from the ten measurements taken on a specimen before testing in accordance with Section 14. The average thickness shall not vary more than ±0.001 in. from the specified nominal thickness, and no individual measurement shall vary more than +0.002 or −0.001 in. for tapes from 5 to 9 mils in thickness, nor more than ±0.003 in. for tapes 10 to 12 mils in thickness, from the specified nominal thickness.

Width

7. The nominal width shall be specified in the purchase order. Individual measurements on all the samples selected under Section 5 shall not vary more than $\pm\frac{1}{32}$ in. from the specified nominal width for tapes 1 in. or less in width, nor more than $\pm\frac{1}{16}$ in. for tapes over 1 in. in width.

Length

8. The nominal length or yards per roll shall be specified in the purchase order. The measured lengths of individual rolls selected under Section 5 shall be not less than that specified.

PHYSICAL PROPERTIES AND TESTS

Surface

9. Tapes are roughly divisible into two classes: "greasy" and "tacky." Greasy tapes shall have the property of sliding freely, one layer upon another, when moved easily between two fingers.

Flexibility

10. The varnish coating shall be flexible and shall not crack when the tape is doubled upon itself and creased between the thumb and

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[1] See p. 1047.

index finger. Care shall be taken not to bring the finger nails into contact with the tape surfaces while making the crease.

Tensile Breaking Strength

11. (*a*) The tensile breaking strength requirement varies with the nominal thickness of the tape. Let t represent the nominal thickness in mils; then, the average tensile breaking strength of the specimens should be not less than $35 + (t-7)$ lb. per inch of width. If the first specimen from a roll does not conform to this requirement, tests of two additional specimens from the same roll shall be made. Then the average of the three tests shall conform to the above requirements. The tensile breaking strength of any of the tests made shall be not less than $29 + (t-7)$ lb. per inch of width.

(*b*) The tensile breaking strength of the "jointed selvages" shall not be less than 80 per cent of the values specified in Paragraph (*a*).

Elongation

12. (*a*) For tapes of nominal width not less than 0.5 in., the elongation of one specimen from each of the selected rolls shall be measured at a temperature T between 20 and 30 C. and shall be not less than that specified in the following table:

NOMINAL THICKNESS	LOAD, LB. PER INCH OF TAPE WIDTH	ELONGATION, PER CENT
7 mils	5	$1.7 + (T-20) \times 0.1$
Over 7 mils	6	$1.7 + (T-20) \times 0.1$

(*b*) The specimens tested for elongation shall be used for electrical tests as described in Section 15.

Resistance to Oil

13. (*a*) One specimen from each of the selected rolls shall be subjected to test for resistance to oil described in the test methods referred to in Section 5. The oil used shall be an insulating oil having a flash point of not less than 135 C. (275 F.), as determined by the Cleveland open cup in accordance with the Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92) of the American Society for Testing Materials.[1] The varnished film shall show no evidence of disintegration in the oil nor flake either in the oil or on the blotter used in the test.

(*b*) The specimens tested for resistance to oil shall be used for electrical tests as described in Section 16.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 892.

Dielectric Strength, as Received

14. (*a*) Ten punctures shall be made on one specimen from each roll as received. The dielectric strength values are the puncture values in volts divided by the average of the ten thickness readings on the specimens in mils. The average of the ten dielectric strength values for each roll as received, shall conform to the requirements shown in Table I. The dielectric strength values when analyzed to determine the uniformity of the material shall conform to the requirements of Paragraphs (*b*) and (*c*).

TABLE I.—DIELECTRIC STRENGTH REQUIREMENTS FOR VARNISHED CLOTH TAPE.

	DIELECTRIC STRENGTH, MINIMUM AVERAGE VOLTS PER MIL		
NOMINAL THICKNESS	AS RECEIVED	AFTER ELONGATION	AFTER HOT OIL
Over 10 mils	1100	1000	1100
9 and 10 mils, incl.	1000	900	1000
Under 9 mils	900	800	900

(*b*) *Uniformity Within the Roll.*—The deviation factor for each roll in each shipment shall be not more than 0.08 when calculated from the following formula:

$$\text{Deviation factor of roll} = \frac{h - l}{h + l}$$

where h = the average of the five highest dielectric strength values of the roll, in volts per mil; and
l = the average of the five lowest dielectric strength values of the roll, in volts per mil.

(*c*) *Uniformity of the Shipment.*—The deviation factor for each roll in each shipment shall be not more than 0.07 when calculated from the following formula:

$$\text{Deviation factor of shipment} = \frac{a - l}{a + l}$$

where a = the average dielectric strength of the entire shipment, in volts per mil; and
l = the average of the five lowest dielectric strength values of the roll, in volts per mil.

Dielectric Strength, After Elongation

15. Five punctures shall be made inside the gage length on each specimen after the elongation test specified in Section 12. The average of the five dielectric strength values for each roll shall conform to the requirements shown in Table I.

Dielectric Strength, After Hot Oil

16. Five punctures shall be made on each specimen after the test for resistance to oil specified in Section 13. The average of the five dielectric strength values for each roll shall conform to the requirements shown in Table I.

PACKING AND MARKING

Packing

17. The purchaser shall specify whether tape is to be supplied dry or immersed in oil.

Dry Packing

18. In the case of dry tape, the purchaser shall specify the number of rolls per package and whether each roll shall be dipped or whether each package shall be dipped in a moisture-resistant wax or neither or both. The wrapping shall be secure and shall protect the contents.

Packing in Oil

19. Oil-immersed tape shall be supplied in suitable oil-tight cans with easily removable lids. The purchaser shall specify the number, width and length of rolls to be supplied in each can. The type of oil used for packing shall be subject to the approval of the purchaser.

Marking

20. The purchaser shall specify the marking desired on each package or can; unless the marking is definitely specified, the package shall conform with the usual trade customs.

INSPECTION AND REJECTION

Inspection and Testing

21. The material shall be inspected and tested within three weeks of its receipt by the purchaser.

Rejection

22. (*a*) The purchaser reserves the right to reject any part of the shipment that does not conform to the requirements for packing and marking as specified in Sections 18, 19, and 20.

(*b*) The failure of a sample roll to conform to the requirements of one or more of the sections enumerated under Paragraphs (*c*) and (*d*) shall be counted as only one roll failure.

(*c*) The failure of 35 per cent or more of the sampled rolls to conform to the requirements of Sections 3, 4, 6, 7, 8, 9, and 10 shall constitute cause for rejection of the entire shipment without further tests.

(*d*) The failure of 50 per cent or more of the sampled rolls to conform to the requirements of Sections 11, 12, 13, 14, 15, and 16 shall constitute cause for rejection of the entire shipment.

NOTE.—The following illustrates a convenient method of recording failures of sampled rolls:

ROLL NO.	FAILURES UNDER SECTIONS							ROLL FAILURES
	3	4	6	7	8	9	10	
1			X					X
2			X	X				X
3								
4								
5								
6				X	X		X	X
7								
8								
9								
10								
Total rolls sampled = 10								Total failures = 3 rolls

Thirty per cent of the rolls failed; therefore, this shipment passes Paragraph (*c*).

Claims

23. Rejection claims shall be made to the seller in writing within four weeks of receipt of material by the purchaser. The results of tests made by the purchaser on which rejection is based shall be furnished to the seller, who shall, within one week of receipt of all such claims, either agree to satisfy the claim or send a representative to the plant of the purchaser to resample the shipment. Samples so selected shall be sealed and submitted to an umpire, mutually agreeable to the purchaser and seller, whose decision should be final.

Settlement of Claims

24. The expense of the umpire tests shall be paid by the loser. In the case of rejection being established, the seller shall pay the freight both ways on the rejected material.

TENTATIVE SPECIFICATIONS
FOR
FLEXIBLE VARNISHED TUBING USED IN ELECTRICAL INSULATION[1]

A.S.T.M. Designation: D 372 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1933; REVISED, 1935, 1937.

Scope

1. These specifications cover three grades of flexible varnished tubing which are used as electrical insulation for leads on electrical apparatus, as follows:

Grade A.—Tubing generally known as "flexible varnished tubing," or "motor and transformer tubing," or "impregnated magneto tubing;"

Grade B.—Tubing generally known as "radio spaghetti;" and

Grade C.—Tubing generally known as "non-flammable tubing" or "flame-resistant tubing."

NOTE.—The term "varnished tubing" as used in these specifications refers to braided cotton sleeving coated or impregnated and coated with lacquer or a combination of lacquer and varnish or other similar coatings.

Material

2. (*a*) The base material of the tubing shall be good quality braided cotton sleeving.

(*b*) The cotton sleeving shall be uniformly treated, and the finished tubing shall be reasonably free from defects such as pimples, blisters, wrinkles, cracks, and similar imperfections.

Color

3. The color desired shall be specified by the purchaser.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

Dimensions

4. The inside diameters of flexible varnished tubing shall conform to the following requirements:

A.S.T.M. STANDARD SIZES FOR FLEXIBLE VARNISHED TUBING.

	INSIDE DIAMETER, IN.				INSIDE DIAMETER, IN.		
SIZE	MAXIMUM	MINIMUM	NOMINAL	SIZE	MAXIMUM	MINIMUM	NOMINAL
No. 0.....	0.347	0.325	0.330	No. 11....	0.101	0.091	0.095
No. 1.....	0.311	0.289	0.294	No. 12....	0.089	0.081	0.085
No. 2.....	0.278	0.258	0.263	No. 13....	0.080	0.072	0.076
No. 3.....	0.249	0.229	0.234	No. 14....	0.070	0.064	0.066
No. 4.....	0.224	0.204	0.208	No. 15....	0.063	0.057	0.059
No. 5.....	0.198	0.182	0.186	No. 16....	0.055	0.051	0.053
No. 6.....	0.178	0.162	0.166	No. 17....	0.049	0.045	0.047
No. 7.....	0.158	0.144	0.148	No. 18....	0.044	0.040	0.042
No. 8.....	0.141	0.129	0.133	No. 19....	0.040	0.036	0.038
No. 9.....	0.124	0.114	0.118	No. 20....	0.036	0.032	0.034
No. 10....	0.112	0.102	0.106				

NOTE.—The minimum inside diameters in this table are the same as the diameters of copper wire for corresponding A.w.g. or B. & S. gage sizes.

Dielectric Strength

5. (*a*) Grade A tubing shall show an average value in the short-time dielectric strength test of not less than 7000 v., and no one of the ten puncturing voltages shall be less than 5000 v.

(*b*) Grade B tubing shall show an average value in the short-time dielectric strength test of not less than 4000 v., and no one of the ten puncturing voltages shall be less than 2500 v.

(*c*) Grade C tubing shall show an average value in the short-time dielectric strength test of not less than 2500 v., and no one of the ten puncturing voltages shall be less than 1500 v.

Heat Endurance

6. The number of hours of baking in the heat endurance test at which first cracking of the tubing occurs when bent through 180 deg. over the rod specified in Section 13 of the Tentative Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (A.S.T.M. Designation: D 350 – 37 T) of the American Society for Testing Materials[1] shall be not less than the following:

Grade A..	96 hr.
Grade B..	72 hr.
Grade C..	48 hr.

Rate of Burning

7. Tubing furnished under these specifications as Grade C shall require at least 45 sec. to burn a length of 2.54 cm. (1 in.).

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[1] See p. 1059.

Commercial Lengths

8. The tubing shall be supplied in straight lengths of 30 or 36 in. or in continuous lengths as may be specified by the purchaser.

Packing

9. Unless otherwise specified by the purchaser, the tubing shall be packed flat in packages conforming to usual trade customs.

Marking

10. Each package shall be marked with the name or trade mark of the manufacturer, the total number of feet, the size and color of the tubing.

Inspection

11. The tubing shall be inspected and tested within two weeks of the date of receipt by the purchaser.

Rejection

12. If the results of any test do not conform to the requirements prescribed in these specifications, that test shall be repeated on two additional specimens from the same lot, each of which shall conform to the requirements specified. If either of these two additional specimens fails, the lot of tubing represented by that specimen may be rejected at the option of the purchaser.

Rehearing

13. Samples of rejected material shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of test, the manufacturer may make claim for a rehearing within that time.

Methods of Testing

14. Flexible varnished tubing shall be sampled, conditioned, and tested in accordance with the Tentative Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (A.S.T.M. Designation: D 350 – 37 T) of the American Society for Testing Materials.[1]

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[1] See p. 1056.

TENTATIVE METHODS OF TESTING SOLID FILLING AND TREATING COMPOUNDS USED FOR ELECTRICAL INSULATION[1]

A.S.T.M. Designation: D 176 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1923; REVISED, 1924, 1925, 1929, 1933, 1934, 1935, 1936, 1937.

Scope

1. These methods cover physical and electrical tests for solid filling and treating compounds for electrical insulation.

NOTE.—Before testing under these methods it may be necessary to degasify (*a*) materials which tend to entrap gases (usually due to high viscosity) and/or (*b*) materials which tend to froth on heating. (See Appendix I.).

PHYSICAL TESTS

General Physical Tests

2. Each of the following tests shall be made in accordance with the method of test applicable, of the American Society for Testing Materials, as indicated:

(*a*) *Melting Point:* Standard Method of Test for Melting Point of Petrolatum (A.S.T.M. Designation: D 127).[2]

(*b*) *Softening Point*, for compounds having no definite melting point: Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (A.S.T.M. Designation: D 36),[3] or Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (A.S.T.M. Designation: D 61).[4] The method used should be indicated in the report.

(*c*) *Flash and Fire Points:* Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).[5]

(*d*) *Evaporation:* Standard Method of Test for Loss on Heating of Oil and Asphaltic Compounds (A.S.T.M. Designation: D 6).[6]

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 942.

[3] *Ibid.*, p. 1098.

[4] *Ibid.*, p. 1103.

[5] *Ibid.*, p. 892.

[6] *Ibid.*, p. 1071.

(*e*) *Viscosity*, Temperature range from melting point to 150 C.: Saybolt Furol, Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).[1]

(*f*) *Penetration Test*, Temperature range from melting or softening point to −25 C.: Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).[2]

Brittleness

3. To determine the brittleness of the material, the compound shall be spread on a piece of sheet metal in a layer $\frac{1}{16}$ in. in thickness. It shall then be submerged in water having a temperature at least 20 F. above the expected breaking point. It shall then be cooled 5 F. and held at this temperature for 5 minutes. The cooling shall then be continued in increments of 5 F. At each temperature interval the compounds shall be tested with the blade of a knife, inserting the point with the blade as nearly parallel with the sheet metal as is practicable, and pulling the compound from the sheet metal as rapidly as possible by rotating the blade about its back as an axis. The temperature at which the compound no longer stretches but snaps, shall be considered the breaking point.

SPECIFIC GRAVITY

Scope

4. This test is intended to determine the specific gravity of both untreated and degasified compounds at any temperature. The approximate volume occupied by entrapped gas at any temperature can be calculated from the difference in specific gravity of an untreated and a degasified sample at that same temperature.

Procedure

5. (*a*) The specific gravity shall be determined by the Standard Method of Test for Specific Gravity of Asphalts and Tar Pitches Sufficiently Solid to be Handled in Fragments (A.S.T.M. Designation: D 71)[3] or by the Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements and Soft Tar Pitches (A.S.T.M. Designation: D 70)[4] of the American Society for Testing Materials.

(*b*) If degasification is required, it shall be performed as described in Section 11 and Appendix I.

Volume of Entrapped Gases

6. The percentage of entrapped gases may be calculated from the following formula:

$$\text{Volume, in per cent, of entrapped gases} = \frac{\text{sp. gr. of degasified compound} - \text{sp. gr. of untreated compound}}{\text{sp. gr. of degasified compound}} \times 100$$

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 982.

[2] *Ibid.*, p. 1080.

[3] *Ibid.*, p. 1108.

[4] *Ibid.*, p. 1110.

COEFFICIENT OF EXPANSION

NOTE.—Certain materials when cooled form voids within the mass and no satisfactory method has been found to measure their coefficient of expansion.

Scope

7. These methods are intended for determining the true and effective coefficient of expansion of compounds. The true coefficient of expansion is for compounds which are free of entrapped gases. The effective coefficient of expansion is the coefficient for material which has not been degasified just prior to test. It is important for many purposes to know the effective coefficient of the material as received or after heating to the maximum temperature of application. Consistent results, however, may only be obtained with gas-free compounds.

Methods

8. The following methods are covered:

Methods A and B.—Methods *A* and *B* for true coefficient of expansion are intended for use only where the uniformity of the material under test justifies a high degree of precision. Method *A* is suitable for testing low-viscosity types such as waxes and petrolatums. Method *B* is suitable for testing asphalts and high-viscosity materials, also for opaque materials which may give difficulty in reading the glass scale of Method *A*.

Methods C, D and E.—Methods *C*, *D* and *E* are intended for faster testing where high precision may not be justified. These methods may be used for determining either true or effective coefficient of expansion.

Method A. Using Glass Flask

Apparatus

9. (*a*) *Flask.*—The flask[1] shall hold approximately 250 ml. to the zero mark, and the neck of the flask shall be 1 cm. in internal diameter and graduated for 25 ml. in 0.1-ml. divisions.

(*b*) *Oil Bath.*—The cylindrical oil bath for heating the sample shall be approximately 10 in. (25.4 cm.) in inside diameter and 20 in. (50.8 cm.) in inside depth with a false bottom 1 in. from the bottom and shall have provision for circulating and heating the oil.

(*c*) *Lead Collar.*—A lead collar shall be hung on the flask during test to prevent the oil currents of the bath from moving the flask.

Calibration

10. The capacity of the flask at the zero point and several points on the scale shall be determined by filling the flask with distilled water at a known temperature and weighing.

Procedure

11. (*a*) The flask shall be maintained under a vacuum of 25 in. of mercury and a temperature 50 C. higher than the softening point

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[1] A Pyrex or quartz glass flask is very satisfactory for this purpose.

(ring-and-ball method, as determined under Section 2 (*b*)) while being filled and for approximately 30 min. after filling is complete. The flask shall be filled to within the last milliliter marked on the neck when held at the maximum test temperature and slowly cooled to room temperature (10 to 12 hr.). Before starting the test, the flask shall be examined for the presence of cavities or irregular contraction of the compound. Some compounds after cooling below the liquid state, tend to stick to the sides of the neck of the flask. In such cases it is necessary to gradually warm the neck and flow the compound to meet the rest, after which the flask shall be placed in the bath for several hours to insure temperature equilibrium.

(*b*) With the compound satisfactorily placed in the flask at the lowest temperature, the height of the column in the neck shall be read and the bath then slowly heated. Readings shall be taken at 50 C. intervals, holding the bath as constant as possible at each point until no more expansion occurs at that point, the procedure being repeated for each point until maximum temperature is reached.

(*c*) *Precautions.*—During the test, temperature readings shall be taken at top and bottom of the bath to detect any variation. Readings of the expansion of the compound shall be made at intervals long enough to insure uniform temperature distribution and complete movement of the compound. Until complete liquefaction, the interval should be 3 to 4 hr.; after liquefaction it may be reduced to 30 min.

Calculation

12. (*a*) After securing the readings over the temperature range desired, a curve shall be plotted from the temperature and expansion readings from which the coefficient of expansion shall be calculated from the following formula:

$$\text{Coefficient of expansion of the compound} = \frac{V_1 - V}{(T_1 - T)\, V} + C$$

where V = the original volume occupied by the compound;
V_1 = the volume at higher temperature occupied by the compound;
T = the original temperature;
T_1 = the higher temperature, and
C = a constant = coefficient of expansion of glass container.

(*b*) The coefficient of expansion shall be calculated for three temperature ranges, as follows:

(*1*) From the minimum temperature at which the measurement was made to 10 C. below the melting point. This is intended to give the average coefficient for the solid condition.

(*2*) From 5 C. above the melting point to 100 C. This is intended to give the average coefficient for the liquid condition.

(*3*) From the minimum temperature at which a measurement was made to 100 C.

Method B. Using Metallic Cell

Apparatus

13. (*a*) *Metal Cell.*—The cell shall be made of steel, consisting of four parts, a cylinder about 2.5 in. in internal diameter, having a rigid bottom, a metallic gasket, a cover to which a steel capillary tube is attached and an auxiliary cover for introducing the compound to be tested while the cell is under a vacuum. The cell shall have an internal volume of approximately 250 ml. A metallic cell that has been found suitable is described in Appendix II.

(*b*) *Oil Bath.*—The oil bath shall be the same as described in Section 9 (*b*), Method *A*, with the exception that provision shall be made for supporting the metal cell.

Calibration

14. The cell shall be calibrated to determine its volume at various temperatures as follows:

(*a*) Weigh the assembled cell to determine its tare weight.

(*b*) Fill the cell with mercury until replacing the cover causes some to extrude through the capillary tubing. Record the weight of the cell and mercury and note the temperature.

(*c*) Place cell in the oil bath in an inverted position. The capillary tubing should extend over the side of the oil bath in such a way that the extruded mercury may be caught in a beaker. The oil bath which is several degrees above room temperature causes some mercury to be extruded from the capillary tube. When all expansion has taken place, weigh the mercury collected.

(*d*) Adjust the oil bath for other test temperatures and note the amounts of mercury extruded. The weight of mercury in the cell at any temperature is thus determined and the volume may be calculated.

Procedure

15. (*a*) The cell shall be fitted with the auxiliary cover and held in an upright position in the oil bath while filling. A vacuum of 25 in. of mercury and a temperature 50 C. higher than the softening point (ring-and-ball method, as determined under Section 2 (*b*)) shall be maintained while filling and for at least 30 min. after the cell has been filled to within approximately $\frac{1}{4}$ in. of the cover. The cell shall then be slowly cooled to room temperature, and any irregularities in the surface of the compound removed.

(*b*) The auxiliary cover shall be replaced with the standard cover and the cell and compound weighed.

(*c*) Sufficient mercury shall be poured into the cell so that some is extruded when the cover is screwed down. The cell shall then be weighed again.

(*d*) The cell shall then be inverted and placed in the oil bath, and the procedure outlined under calibration, Section 14 (*c*) and (*d*) again followed for 5 C. intervals.

(*e*) *Precautions.*—Only clean, distilled mercury shall be used. During the test, temperature readings shall be taken at top and bottom of the bath to detect any variation. Readings of the expansion of the compound should be made at intervals long enough to insure uniform temperature distribution and complete movement of the compound. Until complete liquefaction, the interval should be 3 to 4 hr.; after liquefaction, it may be reduced to 30 min.

Calculation

16. (*a*) After volumetric determinations have been made over the desired temperature range, a curve shall be plotted between volume and temperature readings from which the coefficient of expansion shall be calculated from the following formula:

$$\text{Coefficient of expansion of the compound} = \frac{V_1 - V}{(T_1 - T)\,V}$$

where V = the original volume occupied by the compound;
V_1 = the volume at higher temperature occupied by the compound;
T = the original temperature, and
T_1 = the final temperature.

(*b*) The coefficient of expansion shall be calculated for the same three ranges as prescribed in Method *A*, Section 12 (*b*).

Report

17. The report shall include the following:

(*a*) The type of cell used.

(*b*) A copy of the volume-temperature curve.

(*c*) The temperature ranges as defined under Section 12 (*b*) or 16 (*b*).

(*d*) The coefficient of expansion corresponding to each of the three temperature ranges.

Method C. Specific Gravity Method

Procedure

18. The specific gravity of untreated or degasified compound at two test temperatures may be found by either of the procedures specified in Section 5.

Calculation

19. From the temperature and specific gravity readings the coefficient of expansion shall be calculated from the following formula:

$$\text{Coefficient of expansion} = \frac{\text{sp. gr. at } T - \text{sp. gr. at } T_1}{(T_1 - T)\text{ sp. gr. at } T_1}$$

where T = the initial temperature, and
T_1 = the higher temperature.

Method D. Pycnometer Contraction

Scope

20. This method is a modification of the specific gravity method (Method *C*). It may be applied to either untreated or degasified compounds.

Apparatus

21. (*a*) *Flask.*—A 100-ml. volumetric glass[1] flask shall be used and the zero mark shall be as near as possible to the bulb of the flask.

(*b*) *Oil Bath.*—The oil bath may consist of a tall-form glass[1] beaker of sufficient size so that when the flask is supported about 1 in. from the bottom the oil level will reach at least to the zero mark of the flask.

(*c*) *Metal Collar.*—Lead or iron collars placed on the neck of the flask shall be used during heating to prevent oil currents of the bath from moving the flask.

Procedure

22. (*a*) The flask shall be weighed and then filled to within approximately 10 ml. of the zero mark with the compound to be tested, care being taken that at no time shall the temperature of the compound exceed the softening point (ring-and-ball method) by more than 10 C. Care shall also be taken so that none of the compound remains in the stem of the flask. The flask shall be cooled to room temperature, weighed, immersed in the oil bath and placed in an oven previously heated to the maximum temperature at which the test is to be run. After the oil bath reaches the test temperature it shall be allowed to remain at this temperature for 1 hr. The flask shall be removed from the oven, and mercury from a burette shall be quickly poured into it until the level of the compound is within 0.5 ml. of the zero mark. The quantity of mercury used shall be noted in milliliters. The flask shall be replaced in the oven for 20 min., removed again and the mercury titration shall be continued until the level of the compound is exactly at the zero mark. The number of milliliters of mercury used in both titrations shall be recorded.

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[1] Pyrex glass or other material with equally low coefficient of expansion is very satisfactory for this purpose.

(*b*) The oven temperature shall be reset at the next lower test point, temperature equilibrium allowed for as above, and the titration shall be repeated. The same procedure shall be continued at all subsequent test points, the milliliters of mercury added at each point being recorded.

Calculations

23. To find the coefficient of expansion of the material between any two temperatures, the calculations shall be made as follows:

$$\text{Volume of mercury } A \text{ at room temperature corrected to temperature } t = A + 0.000182A\,(t - 20)$$

Example.—If volume of flask is 100 ml. at 20 C.; volume at $t = 100 + 0.0025\,(t - 20)$ ml.

$$\text{Let } X = V_1 - M_1 = \text{the volume of compound at } T_1,$$
$$\text{Let } Y = V_2 - M_2 = \text{the volume of compound at } T_2,$$

Then the expansion in cu. cm. per gram, $T_1 - T_2 = \dfrac{X}{W} - \dfrac{Y}{W}$

the expansion per cu. cm., $T_1 - T_2 = \dfrac{\dfrac{X - Y}{W}}{\dfrac{Y}{W}} = \dfrac{X - Y}{Y}$

the coefficient of expansion, cu. cm. per cu. cm. per degree Centigrade. $= \dfrac{X - Y}{Y\,(T_1 - T_2)}$

where W = the weight of sample in grams
T_1 = the higher temperature in degrees Centigrade
T_2 = the lower temperature in degrees Centigrade
M_1 = milliliters of mercury (corrected) added at T_1
M_2 = the sum of milliliters of mercury (corrected) added at T_1 and at T_2
V_1 = the corrected volume of flask at T_1
V_2 = the corrected volume of flask at T_2

To obtain the curve of expansion, plot the points $\dfrac{W}{X}$, $\dfrac{W}{Y}$, etc., which are the densities at various temperatures.

Method E. Pycnometer Expansion

Scope

24. This method is another modification of the specific gravity method (Method *C*) and may also be applied to either untreated or degasified materials.

Apparatus and Calibration

25. The pycnometer and bath required are the same as described in Section 21, except that the neck of the flask shall be cut off at the 100-ml.

point and ground square. A metal pycnometer may be used provided its coefficient of expansion is known and is used in Section 27.

Procedure

26. In this procedure the pycnometer shall be allowed to cool slowly to the lowest test temperature. During the cooling period the flask shall be kept filled by adding more compound and after equilibrium is reached the excess material shall be removed by passing a sharp, flat blade over the rim. The flask shall then be removed from the bath and quickly weighed. Knowing the tare weight and volume of the flask, the specific gravity may be determined. For successively higher temperatures, it is only necessary to weigh the extruded portion.

Calculations

27. From the temperature and weight readings the coefficient of expansion may be calculated from the following formula:

$$\text{Coefficient of expansion} = \frac{W - W_1}{W_1(T_1 - \mathrm{T})} - \frac{WC}{W_1}$$

where W = the initial weight of the compound in the flask
W_1 = the weight of the compound in the flask at higher temperature
T = the initial temperature
T_1 = the higher temperature
C = the coefficient of expansion of the flask

Method F. Plummet Displacement

Scope

28. This method is another modification of the specific gravity method (Method C).

Apparatus

29. The apparatus shall consist of the following:

(*a*) *Balance.*—An analytical balance equipped with pan straddle.
(*b*) *Plummet.*—An aluminum plummet of suitable shape weighing 5 to 10 g.
(*c*) *Beaker.*—A 400-ml. glass[1] beaker wrapped with asbestos.
(*d*) *Thermometer.*—A thermometer of suitable range.
(*e*) *Wire.*—Two pieces of fine copper wire.

Procedure

30. The specific gravity at pouring temperature shall be calculated from the weight of compound displaced by the calibrated aluminum plummet.

31. (*a*) *Calibration of Plummet.*—The following weight determinations of the plummet, to the nearest 1 mg., shall be made:

a = the weight in air;
b = the weight suspended in water at 25 C. (77 F.).

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[1] Pyrex glass or other material with equally low coefficient of expansion is very satisfactory for this purpose.

Then: $a - b$ = the weight of water displacement at 25 C. (77 F.).

The value of the plummet displacement (D_{t_p}) in terms of grams of water at 25 C. (77 F.) shall then be corrected to the pouring temperature, t_p, in Centigrade degrees by means of the following formula:

$$D_{t_p} = 0.000076^{a}\,(t_p - 25)\,(a - b) + (a - b)$$

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[a] Coefficient of cubical expansion of aluminum per deg. Cent.

(*b*) *Testing of the Sample.*—The sample shall be carefully melted in the beaker and the temperature raised to approximately 15 C. (27 F.) above the desired test temperature. The beaker shall be placed on the straddle and the plummet suspended in the compound by the fine copper wire. (The weight of the wire should be balanced by placing an equal weight of the wire on the opposite balance pan.)

The scales shall be balanced approximately and at the same time the sample shall be stirred slowly, using the thermometer as a stirring rod. When the sample has cooled to the desired temperature, the weighing shall be completed rapidly.

(*c*) *Calculation of Specific Gravity, t_p/25 C.*—The specific gravity shall be calculated from following formula:

$$\text{Sp. Gr., } t_p/25 \text{ C.} = \frac{\text{Weight of Plummet in Air} - \text{Weight of Plummet in Compound}}{D_{t_p}}$$

Calculation of Coefficient of Expansion

32. After the specific gravity of the sample at 25 C. (77 F.), and at pouring temperatures have been determined according to the above described methods, the coefficient of expansion shall be calculated according to the formula given in Section 19.

ELECTRICAL TESTS

DIELECTRIC STRENGTH TEST

Scope

33. This test is intended to determine the short-time dielectric strength of solid filling and treating compounds. It is of value for purposes of comparison or as an indicator of the condition of a compound, but it is not a measure of the dielectric strength of compounds when subjected to prolonged stresses.

Apparatus

34. The equipment used for producing, controlling, and measuring voltage shall conform to that prescribed in Section 2 of the Tentative Methods of Testing Sheet, Tape, and Molded Insulating Materials for Dielectric Strength (A.S.T.M. Designation: D 149 – 36 T) of the American Society for Testing Materials.[1]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 918 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1002.

Electrodes

35. The compound shall be tested between polished hemispherical electrodes ½ in. in diameter separated by a gap of 0.1 in.

NOTE.—A convenient form of apparatus for holding the electrodes and compound is described in Appendix III.[1] It is recognized that when liquid materials are being tested, this method is inconsistent with the Standard Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation: D 117) of the American Society for Testing Materials.[2] However, since spherical electrodes are considered preferable for solid and semisolid materials, the advantage of using spherical electrodes, in order that direct comparison can be made in both the liquid and solid states, is sufficiently great to justify the inconsistency with Methods D 117.

Procedure

36. (*a*) A representative sample of the material shall be taken from the original package. This sample shall be melted and poured directly into the testing container, care being taken not to overheat the compound nor to entrap air. A melting and pouring temperature of approximately 50 C. above the softening point (ring-and-ball method) as determined under Section 2 (*b*) is recommended. Pasteboard test receptacles shall be thoroughly dried out by heating before using, and the entire test container assembly shall be heated to its safe maximum temperature (85 to 90 C. if bakelite is used) before the compound is poured into it.

(*b*) The testing container, after filling, shall be placed in a suitable temperature control chamber and left for 4 hr. or until the compound has been brought uniformly to the desired test temperature. The temperature may be determined by a thermocouple inserted along the axis of one of the electrodes to a point near the tip.

(*c*) Voltage shall then be applied to the test specimen at a uniform rate of increase of 1000 v. per sec. from zero to breakdown.

(*d*) Specimens shall be tested in air unless the breakdown voltage exceeds 100 kv. If the breakdown voltage is greater than 100 kv., the test container shall be immersed in clean, dry oil. Before the specimen is immersed, the oil shall be brought to the test temperature. Care shall be taken not to contaminate the compound with the oil.

(*e*) Variations of gap setting with temperature may be determined by varying the temperature of the test assembly when empty. For well-designed test apparatus, these variations are so small as to be negligible. For maximum accuracy, or for cases where the variation might be appreciable, a temperature correction curve for the gap may be determined and allowance made for gap variation when a test setting is made.

Number of Tests

37. (*a*) As the dielectric strength of filling and treating compounds varies widely with temperature, it is recommended that tests be made at a sufficient number of temperatures to establish the dielectric strength - temperature curve from 0 to 100 C. For this purpose, tests should be made at 0, 30, 70, and 100 C.

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[1] See p. 1005.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 1167.

(*b*) Five specimens shall be tested at each temperature and the average value taken as the short-time dielectric strength of the compound at that temperature.

Report

38. The report shall include the following:

(*a*) The short-time dielectric strength - temperature curve, and

(*b*) The individual values of the breakdown voltage.

VOLUME RESISTANCE – TEMPERATURE CHARACTERISTICS

Volume Resistance versus Temperature

39. (*a*) This test is intended to determine the volume resistance - temperature characteristics of solid filling and treating compounds. It is of value as a comparative test and also may be used in determining the volume resistivity on an ohm-centimeter basis.

Temperature Range

(*b*) The volume resistance of this class of material varies with the temperature, generally decreasing rapidly with increase of temperature. It is, therefore, recommended that a sufficient number of tests be made at different temperatures to establish the volume resistance – temperature curve. To be of most significance, the test shall include determinations made up to the highest service temperatures. At room temperatures and below, the volume resistance of practically all of these compounds is so high that it cannot be measured with the ordinary galvanometer equipment.

Conductivity Cell

40. A suitable conductivity cell consisting of parallel planes, concentric cylinders, or coaxial cones shall be used in determining the volume resistance of the compound. The distance between electrodes shall not be less than 0.75 mm. (0.03 in.) nor more than 5 mm. (0.2 in.). The voltage gradient shall not exceed 200 v. per millimeter (5 v. per mil). The area of the electrode shall be sufficiently large so that the current flow can be measured, with the apparatus available, to an accuracy within 5 per cent. Electrode areas of 50 to 500 sq. cm. (7.7 to 77.5 sq. in.) should prove suitable. Because of possible catalytic or corrosion effects of some compounds on certain metals, the electrodes should be nickel, gold or platinum plated.

The insulating material used to support the electrodes shall be capable of withstanding the wide temperature range to which the cell is subjected and preferably shall be of an inorganic material such as a ceramic material or suitable glass.

A test run on the empty cell shall be made over the temperature range chosen in order to determine if any leakage is present.

A conductivity cell that has been found suitable is described in Appendix IV.

Sample

41. (*a*) A representative sample shall be taken from the original package, melted and poured directly into the testing container. Care shall be exercised in melting and pouring the compound not to overheat nor to entrap air in it. A melting and pouring temperature of approximately 50 C. above the softening point (ring-and-ball method) as determined under Section 2 (*b*) is recommended. The quantity of the sample depends upon the capacity of the resistivity cell used, but in any case it shall be sufficient to permit three separate determinations. Before filling, the conductivity cell shall be heated to slightly above the pouring temperature of the compound.

(*b*) A suggested procedure in filling the cell, especially in the case of the higher melting compounds, is to determine the quantity of compound necessary to just fill the cell with the electrodes in position. In the case of coaxial cones or concentric cylinders the proper quantity of the heated compound shall be first poured slowly into the outer cylinder. Any bubbles which may form on the surface of the compound shall be removed by a quick application of a flame from a bunsen burner. Immediately the inner electrode shall be slowly lowered into the compound and a thermometer placed in the well.

Test Procedure

42. The conductivity cell, after filling, shall be placed in an oil or air bath having suitable temperature control and sufficient time allowed to elapse to bring the bath and cell to temperature equilibrium at each test temperature. The temperature of the cell shall be determined by two mercury thermometers placed in contact with the electrodes. The temperature of the bath shall be determined by a mercury thermometer placed near the cell. The temperature of the bath shall be within 1 C. of the test temperature when readings are taken. The temperatures of the cell thermometers shall agree within 0.5 C. of each other and within 1 C. of the bath temperature when readings are taken.

Measurements and Calculations

43. The volume resistance shall be measured at each temperature in accordance with the Standard Methods of Test for Resistivity of Insulating Materials (A.S.T.M. Designation: D 257) of the American Society for Testing Materials,[1] with particular attention to Sections 8, 9, 10, 17, 18 and 19 for liquid insulating materials.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1203.

Report

44. The report shall include the following:

(*a*) The type of resistivity cell used;

(*b*) The distance between guarded and unguarded electrodes;

(*c*) The area of guarded electrode;

(*d*) The battery voltage; and

(*e*) A curve plotted from the readings obtained over the temperature range chosen showing the relation of volume resistance in ohm-centimeter units and the temperature in degrees Centigrade.

NOTE.—In lieu of the information required in Paragraphs (*b*) and (*c*), a statement of the approximate overall accuracy may be made.

POWER FACTOR AND DIELECTRIC CONSTANT

Scope

45. This test is intended to determine the power factor and the dielectric constant of solid filling and treating compounds at specified temperatures corresponding to those temperatures to which the materials are to be subjected in service.

Temperature Range

46. Since the power factor and dielectric constant of this class of materials vary with the temperature, a sufficient number of tests shall be made at different temperatures to establish the dielectric constant - temperature curve and the power factor - temperature curve over the temperature range to which the respective material is subjected in service. Determinations shall be made at not less than three temperatures; a temperature near the lowest to which the material is to be subjected in service, ordinary room temperature of 20 to 25 C., and a temperature near the maximum temperature to which it is subjected in service.

NOTE.—For many materials the power factor - temperature curve may have a maximum, minimum, or other irregularity within the working range of temperatures. With such a material many determinations may be required to establish the curve.

Test Specimens

47. The materials to which these tests apply fall into two classes as regards the most suitable type of specimen to be used in the measurement of power factor and dielectric constant. One class includes waxes and other materials which can be cast or pressed into sheet form and then measured between electrodes, such as are used in making tests on porcelain, molded materials and sheet materials. (See the Tentative Methods of Testing Electrical Insulating Materials for Power Factor and Dielectric Constant (A.S.T.M. Designation: D 150 - 36 T) of the American Society for Testing Materials.[1]) The

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 955 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1069.

other class includes materials that are fluid at ordinary temperature or can be made so by increasing their temperatures. Materials in the fluid state can be poured into a measuring cell such as described in Section 52 and the determinations made from which the power factor and the dielectric constant can be calculated.

Measuring Cell

48. In the measuring cell the distance between the electrodes and the area of the electrodes shall be such that a capacitance of not less than 100 $\mu\mu$f. will be obtainable. The voltage gradient shall not be over 5 v. per mil. Due to the catalytic or corrosive effects of some compounds on certain metals, the electrodes may need to be nickel, gold, or platinum plated. The insulating material used to support the electrodes shall be capable of withstanding the wide temperature range to which the cell or material is to be subjected during the tests without any appreciable effect upon the determinations being made. Certain ceramic materials, special glasses, and quartz have been found suitable for insulating supports for the electrodes between which the material is to be measured. One cell that has been found satisfactory for these determinations is described in Appendix IV. Cells of different construction from the one described have been used with equally satisfactory results.

Sampling

49. A representative sample shall be taken from the original package, melted if necessary, and then either cast in the form of a disk or poured directly into the measuring cell. The use of the cell or the disk will depend upon the particular material. In heating and pouring the compound, care shall be exercised not to entrap air in the compound between the electrodes. A melting and pouring temperature of approximately 50 C. above the softening point (ring-and-ball method, as determined under Section 2 (*b*)) is recommended. A sufficient quantity of the material shall be taken as the sample to enable making at least three determinations. Before pouring the compound into the container or cell, the container or cell shall be heated to a temperature slightly higher than the temperature at which the compound is being poured.

Temperature Bath

50. The specimen as prepared above, shall be placed in an oil or air bath having suitable temperature control, and sufficient time allowed to elapse to bring the bath and specimen to the desired temperature equilibrium at each test temperature. The temperature

of the electrodes and the compound shall be determined by two mercury thermometers, or two thermocouples with cold junctions placed in contact with the electrodes. The temperature of the bath shall be determined by a thermometer, or a thermocouple with cold junction placed near the cell or the specimen. The temperature of the bath shall be within 1 C. of the desired test temperature when the determinations are made. The temperature of the specimen thermometers or thermocouples shall agree within 0.5 C. of each other and within 1 C. of the bath temperature when the readings are taken.

Measurements

51. Determinations shall be made of the power factor and dielectric constant at the temperatures and frequencies desired in accordance with the methods outlined for 25 to 60 cycles, 1000 cycles and 100 to 1500 kilocycles as described in the Tentative Methods of Testing Electrical Insulating Materials for Power Factor and Dielectric Constant (A.S.T.M. Designation: D 150 - 36 T) of the American Society for Testing Materials.[1] Measurements shall be made at the frequencies to which the material will be subjected in service unless otherwise specified. When using a cell in making power factor and dielectric constant determinations, measurements shall be made at the temperature desired, first with air as dielectric and then with the compound in the cell as dielectric.

Calculations

52. The power factor shall be calculated in accordance with the Tentative Methods D 150 - 36 T,[1] and the dielectric constant shall be determined for any given compound from the ratio of the capacitance of the cell with the compound as dielectric to the capacitance of the cell with air as dielectric. When the specimen under determination is in the form of a disk the air capacitance shall be calculated from the dimensions of the electrodes and the thickness of the dielectric. In this case, correction shall be made as outlined in the Tentative Methods D 150 - 36 T.[1] When the sample has been poured between electrodes being held apart by separators, the air capacitance can be measured directly the same as with the cell, or it may be calculated from the dimensions of the electrodes and the thickness of the separators, taking into account the edge correction.

Report

53. The report shall include the following:

(*a*) The type of cell used and the dimensions of the same.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 955 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1069.

(*b*) The method used and the frequency at which measurements were made.

(*c*) The voltage across the specimen during the test.

(*d*) The dielectric constant, the power factor, and the respective temperatures at which the determinations were made.

(*e*) A statement of the approximate accuracy of the determinations.

APPENDIX I

DEGASIFICATION OF SOLID FILLING AND TREATING COMPOUNDS

Compounds having high viscosity may be degasified by heating in a vacuum oven or in a cell fitted with a special cover with vacuum connection similar to the cover shown in Fig. A1, Appendix II.

In either method the temperature and vacuum should be high enough and the time long enough to insure the driving off of mechanically entrapped gases, but should tend to decompose the compound as little as possible. A gas-free condition is indicated when bubbles of gas no longer appear at the surface of the compound.

APPENDIX II

CELL FOR DETERMINING COEFFICIENT OF EXPANSION OF SOLID FILLING AND TREATING COMPOUNDS

Figure A1 shows the metallic cell for coefficient of expansion determinations. The cell consists of four principal parts: A steel cylinder, a metallic gasket, a steel cover, and a dummy or auxiliary cover for filling. The gasket must be of a metal which does not amalgamate with mercury.

The cylinder is about 2.5 in. (6.35 cm.) in internal diameter, and approximately 3 in. (7.6 cm.) in internal depth. The top of the cylinder is threaded to receive the steel cover and has a machined shoulder to seat a 0.003 in. thick metallic gasket. The cylinder may be of one piece construction or fitted with a cap at the bottom similar to the top end.

The steel cover is carefully rounded on the under side to avoid air pockets. It is threaded into the top of the cylinder and seats on the metallic gasket. The center of the cover is threaded to receive a steel capillary tube of 0.018 in. in internal diameter.

The auxiliary cover is brass with a rubber gasket seated by atmospheric pressure. A brass funnel and vacuum connection are soldered to the cover. A plug is provided for sealing the funnel while the cell is maintained under the vacuum specified after filling.

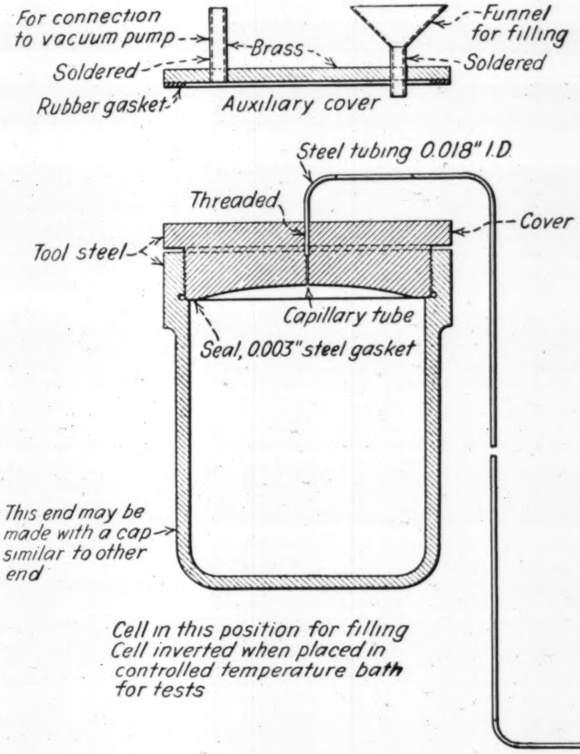

For connection to vacuum pump
Brass
Funnel for filling
Soldered
Soldered
Rubber gasket
Auxiliary cover
Steel tubing 0.018" I.D.
Threaded
Cover
Tool steel
Capillary tube
Seal, 0.003" steel gasket
This end may be made with a cap similar to other end
Cell in this position for filling
Cell inverted when placed in controlled temperature bath for tests

FIG. A1.—Metallic Cell for Coefficient of Expansion Determinations.

APPENDIX III

DEVICE FOR DETERMINING DIELECTRIC STRENGTH OF SOLID FILLING AND TREATING COMPOUNDS

Because of the great difficulty in removing most solid filling and treating compounds from the container, it is desirable to use a test assembly having an inexpensive container which can be thrown away after a test. A device of this sort is illustrated in Fig. A2.

The test assembly consists of a framework made from laminated phenol fiber, large enough to hold loosely a box of heavy paper or pasteboard 10 by 1¾ by 1¾ in. in inside dimensions, with brass bushings centrally inserted in each end piece to hold the electrode rods. The electrodes, which are separable by means of screw joints, are inserted through small holes in the ends of the paper box and clamped to make a compound-tight joint. For the electrodes, a metal is selected that will give minimum gap changes with temperature. Steel has been found quite satisfactory. The proper electrode spacing may be obtained by means of an adjusting screw on the right-hand end.

After use, the electrode supporting screws are backed off; the pasteboard container, holding part of the electrodes, is then easily withdrawn. The electrode parts are salvaged by melting the compound, and the used pasteboard container discarded.

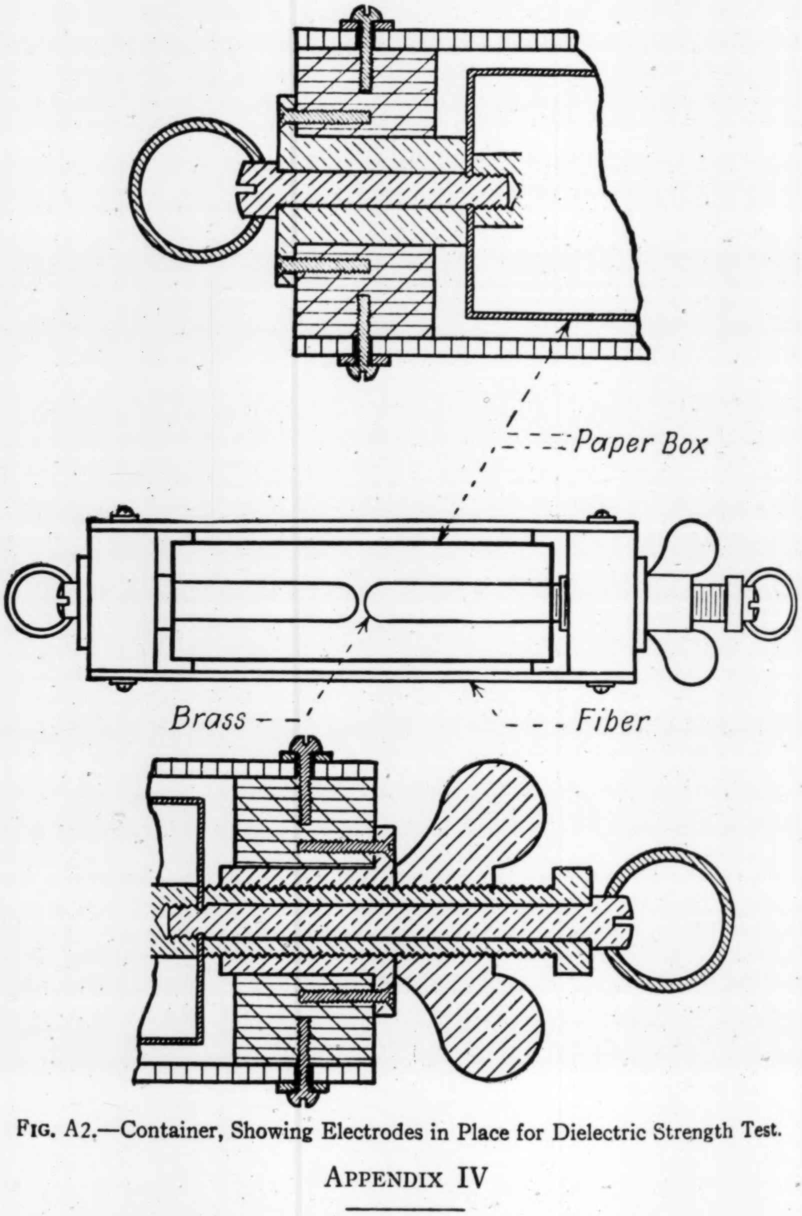


FIG. A2.—Container, Showing Electrodes in Place for Dielectric Strength Test.

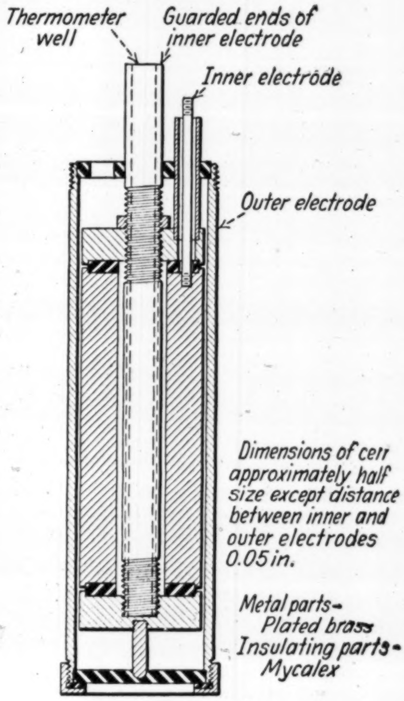
APPENDIX IV

MEASURING CELL FOR RESISTIVITY, POWER FACTOR AND DIELECTRIC CONSTANT MEASUREMENTS OF SOLID FILLING AND TREATING COMPOUNDS

A conductivity cell that has been developed for the purpose of measuring the volume resistivity as well as other electrical constants of solid filling and treating compounds and oil is shown in Fig. A3. The concentric cylinder type of cell was chosen after experimenting with various types for the following points of simplicity and efficiency:

1. Ease of insulating electrodes.
2. Large area of electrodes in compact form, the outer one of which also serves as container for the compound. This outer electrode is also exposed directly to the heating medium and aids in rapid changes from one temperature to another and also promotes uniform temperature distribution to the comparatively thin layer of compound in contact with it.
3. Comparative ease in assembling and disassembling cell and cleaning.

This type of cell permits the use of a very small amount of insulating material to insulate the electrodes. Mycalex has been found to be a satisfactory material for this purpose due to the fact that it can be machined to size and has good insulation resistance characteristics over the temperature range at which these compounds are normally tested. Also, it apparently is unaffected by the solvents used in the cleaning operation.



Dimensions of cell approximately half size except distance between inner and outer electrodes 0.05 in.

Metal parts- Plated brass
Insulating parts- Mycalex

FIG. A3.—Apparatus for Volume Resistance - Temperature Characteristics Test.

A suggested method of cleaning the cell is to remove the bottom retaining ring and to hang the cell in an oven by a hook fastened through a hole drilled in the protruding stem. Upon heating, the outer cylinder will slide away from the inner electrode and both parts will drain fairly clean of the compound. In order to speed up this cleaning, the flame of a bunsen burner may be applied directly to the cell after removing the cap and suspending the cell by the protruding stem. Further application of the flame to the cylinder and inner electrode after they separate will rapidly remove most of the compound and the final cleaning of each part can be accomplished by suitable solvents.

TENTATIVE METHODS OF TESTING PIN-TYPE, LIME GLASS INSULATORS[1]

A.S.T.M. Designation: D 468 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society **is** subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1937.[2]

Scope

1. These methods are intended to apply to the testing of pin-type, lime glass insulators used for primary and secondary power distribution.

SAMPLING

Sampling

2. (*a*) Sampling for purpose of these test methods shall be as specified in the following Paragraphs (*b*) and (*c*). The insulators shall be selected in such a manner as to be representative of the lot. Sampling for testing by the purchaser on individual shipments shall be by mutual agreement between the purchaser and the seller.

(*b*) Every insulator shall be checked during production for the presence or absence of structural defects. One insulator in each fifty shall be gaged. Thermal shock tests and mechanical strength tests shall be made on at least four insulators selected at random every 24 hr. during production.

(*c*) For the electrical flashover and dielectric strength tests (Section 8) specimens representative of the product shall be tested and certified by a commercial or institutional testing laboratory.

ROUTINE TESTS

3. Routine tests consist of visual inspection for physical and structural defects; gaging for pin-hole formation; thermal shock test for homogeneity and uniformity of annealing; and, mechanical test for structural strength, applied in that order.

Structural Defects

4. The finish of the insulators as indicated by the presence or absence of structural defects, such as cracks, chips, folds and stones, shall be determined by visual inspection.

Chips are defects caused by the fracture and complete removal of a portion of the insulator.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

[2] Accepted for publication as tentative by Committee E-10 on Standards, August 26, 1937.

Folds are a linear indentation of the surface of the insulator caused by the meeting and partial welding of a reentrant portion of a surface which has been partially chilled in some previous stage of the process of manufacture.

Stones are foreign inorganic matter completely or partially surrounded by glass usually consisting of particles of refractory material, crystalline products due to combination of refractories and glass batch constituents, particles of incompletely melted batch, or products of devitrification of the glass.

Gaging Threads

5. The gaging test is intended to determine whether the pin hole is properly formed with smooth and uniformly pitched threads. The insulator shall be screwed home on the gage specified (see Appendix I), and when so attached observation shall be made of the clearance in inches between the top end of the gage and the crown of the insulator. There shall be no perceptible play or rocking of the insulator when screwed home on the gage. The number of turns necessary to release the gage shall be determined by holding the insulator upside down and counting the revolutions of the insulator required before it can be disengaged from the gage.

Thermal Shock

6. (*a*) The thermal shock test consists in transferring the insulators from a hot-water bath to a cold-water bath and then from a cold to a hot bath. The hot to cold shock indicates primarily homogeneity, while the cold to hot shock shows up surface imperfections.

(*b*) *Apparatus.*—Two water baths are required, each of which shall be fitted with separate water inlets and drains. One bath shall be equipped with a heating element. The heating supply shall be so placed that the bath will be heated uniformly. Standard thermometers preferably partial-immersion thermometers graduated in degrees Fahrenheit and conforming to the Standard Specifications for A.S.T.M. Partial-Immersion Thermometers for General Use, −20 to +150 C., 0 to +300 F. (A.S.T.M. Designation: D 182) of the American Society for Testing Materials[1] shall be available for checking the temperatures of the baths.

(*c*) *Procedure.*—The water shall be stirred and then the temperature of each of the water baths shall be checked for uniformity by noting the temperature at various places in the bath. The insulators shall be submerged in the bath by means of wooden racks and in such a position as to permit the filling of the pin cavity and between petticoats. At least four insulators of each type shall be tested at one time, half without pins and half with standard pins, of the type to be used, screwed tightly into the pin holes. The pins shall have well-formed threads, and when threads show wear the pins shall be discarded.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1403.

(*d*) *Hot to Cold Shock.*—The insulators shall be placed in the hot-water bath at 125 F. for 10 min. and then changed within 5 sec. to the cold-water bath at 55 F., left therein for 10 min., and then examined.

(*e*) *Cold to Hot Shock.*—The same insulators shall then be placed in the cold-water bath at 55 F. for 10 min. and then changed within 5 sec. to the hot-water bath at 155 F., left therein 10 min., and then examined.

(*f*) *Observations.*—After each of the above thermal shock tests (Paragraphs (*d*) and (*e*)) the insulators shall be examined for presence or absence of cracking, chipping and spalling.

Mechanical Strength

7. The insulators shall be mounted on a lead-capped, steel pin of sufficient strength to prevent material deflection within the limits of the pull required to fail the insulator. The load shall be applied mechanically at right angles to the pin through a flexible wire rope in the groove of the insulator. The load shall be applied uniformly and the speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. per min. when the machine is running idle. The strength of the insulator shall be the total load at first visible crack or break of the glass.

ELECTRICAL TESTS

Electrical Flashover and Dielectric Strength

8. Electrical flashover and dielectric strength tests when applied shall be made in accordance with the tests for Pin Insulators as described in the American Institute of Electrical Engineers' Standard for Insulator Tests (A.I.E.E. No. 41, March, 1930) (American Standard C 29a–1930).

NOTE.—The applicable sections of the A.I.E.E. standard are given in Appendix II of these methods.

APPENDIX I

INSULATOR THREADS AND GAGES

This appendix is incorporated as a guide in guarding against the common contingency of misunderstanding by the use of improper gages occasioned by the fact that pin formations for insulators used by the power industry are different from the pin formations for insulators used by the telephone and telegraph industry. The insulator gages and thread formations shown in Fig. 1 are those adopted as standard for the power industry by the Edison Electric Institute and form part of its standard entitled "Suggested Specifications for Insulators and Pin Threads and Gages," approved by the Transmission and Distribution Committee, Edison Electric Institute (TD-71, 1937).

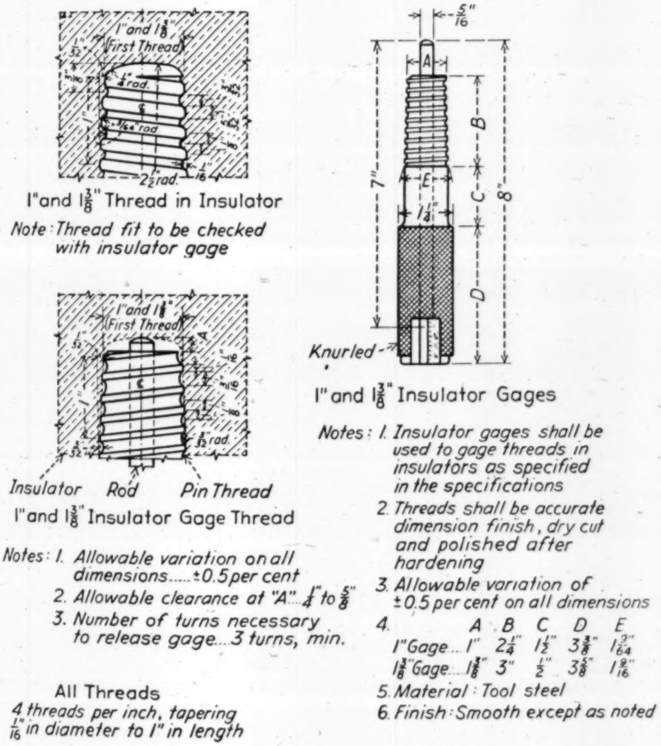
1" and 1⅜" (First Thread)
¼" rad.
3/64" rad.
2½" rad.
1" and 1⅜" Thread in Insulator
Note: Thread fit to be checked with insulator gage
1" and 1⅜" (First Thread)
A
3/32" rad.
Insulator
Rod
Pin Thread
1" and 1⅜" Insulator Gage Thread
Notes: 1. Allowable variation on all dimensions.....±0.5 per cent
2. Allowable clearance at "A"....¼" to ⅝"
3. Number of turns necessary to release gage....3 turns, min.
All Threads
4 threads per inch, tapering 1/16" in diameter to 1" in length
5/16"
A
B
E
C
1¾"
D
7"
8"
Knurled
1" and 1⅜" Insulator Gages
Notes: 1. Insulator gages shall be used to gage threads in insulators as specified in the specifications
2. Threads shall be accurate dimension finish, dry cut and polished after hardening
3. Allowable variation of ±0.5 per cent on all dimensions
4. A B C D E
1" Gage... 1" 2¼" 1½" 3⅜" 1 9/64"
1⅜" Gage... 1⅜" 3" ½" 3⅝" 1 9/16"
5. Material: Tool steel
6. Finish: Smooth except as noted

FIG. 1.—Insulator Threads and Gages.

APPENDIX II

ELECTRICAL FLASHOVER AND DIELECTRIC STRENGTH TESTS

The following descriptions of the tests for electrical flashover and dielectric strength have been taken from the American Institute of Electrical Engineers' Standard for Insulator Tests (A.I.E.E. No. 41, March, 1930) (American Standard C 29a-1930), Paragraphs 41-100, 41-101, 41-103, 41-104, 41-202, 41-203, 41-600 and 41-601. Sections A6 and A7 appear in the A.I.E.E. standard as Paragraphs 41-600 and 41-601 in an appendix entitled "for information only."

A1. *Dry Flashover Test.*[1]—Dry flashover test shall be performed with the pin insulator mounted in a vertical position on a steel pin of circular section 1 in. (2.54 cm.) in diameter mounted on a cross-arm, and of such length that the ratio of the shortest distance from the outer edge of the head, around the insulator to the cross-arm, to the shortest distance from the edge of the head around the insulator to the pin shall be 1.25. The cross-arm shall be a grounded metallic tube not less than 3 in. (7.63 cm.) and not more than 5 in. (12.7 cm.) in outside diameter and shall extend at least 3 ft. (0.914 m.) on either side of the center line of the insulator pin. No other grounded structure shall be nearer than 3 ft. (0.914 m.) to any part of the insulator or conductor. The head of the insulator shall be fitted with a straight smooth metallic rod or tube not less than 0.5 in. (1.27 cm.) in outside diameter extending in a direction at right angles to the cross-arm and at least 2 ft. (0.609 m.) in either direction from the center line of the insulator head. This rod shall be secured in the upper groove by means of at least one turn of wire not smaller than No. 8 A.w.g. placed in the side tie wire groove, the ends being closely wrapped about the rod on each side of the insulator out to a point vertically above the edge of the largest petticoat.

The test shall be performed by applying voltage between the rod fastened to the head and the steel pin, and raising it at a rate of approximately 10,000 v. every 15 sec. to a value at which dry flashover occurs. The initial applied voltage may be raised quickly to approximately 80 per cent of dry flashover voltage.

Flashover voltage shall be determined at or corrected to a standard humidity corresponding to a vapor pressure of 0.6085 in. (15.45 mm.) of mercury. This is equivalent to a relative humidity of 65 per cent at 77 F. (25 C.) and a barometric pressure of 30.0 in. (76.2 cm.) of mercury. Humidity shall be measured with wet and dry bulb thermometers, the air being circulated past the thermometers at a velocity of 3 m. (9.84 ft.) or more per second or by the sling psychrometer. The measurements shall be reduced to vapor pressure with the assistance of the Smithsonian Meteorological Tables or by the following formula:

$$E = E' - 0.000367\,B\,(t - t')\left(1 + \frac{t' - 32}{1571}\right)$$

where E = vapor pressure in inches,
E' = pressure of saturated aqueous vapor at temperature t',
t = temperature of the air in Fahrenheit degrees,
t' = temperature of the wet-bulb thermometer in Fahrenheit degrees, and
B = barometric pressure in inches.

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[1] It is expected that the data obtained from these tests will permit development of a practicable laboratory procedure for correcting flashover tests for humidity. (See Paragraphs A6 and A7.)

A2. *Wet Flashover Test.*—Except that no provision need be made for controlling the temperature and relative humidity of the air, the testing arrangement shall be the same as in the dry flashover test with the addition of equipment to provide a finely divided and reasonably uniform spray at an angle of 45 deg. from the vertical and at a rate of precipitation of 0.2 in. (5.07 mm.) per min. The water shall have a resistance of from 6000 to 8000 ohms per inch cube (15,200 to 20,300 ohms per cm. cube) and shall be delivered to the spray nozzle at a pressure of not less than 35 and not more than 50 lb. per sq. in. (2.46 to 3.51 kg. per sq. cm.) measured at the nozzle. The vertical and horizontal dimensions of the vertical area sprayed shall be measured in a plane through the vertical axis of the insulator and shall be at least 1.75 times the corresponding over-all projected dimensions of the insulator. The precipitation shall be determined by measurements taken, with the insulator removed, at the location of the top, center and bottom of the vertical axis of the insulator when in its test position. The water shall be collected in a vessel having a top diameter of 6 in. (15.24 cm.). Individual measurements shall show a variation of not more than 25 per cent from the mean of the three measurements.

This standard spray shall be applied to the insulator for 1 min. prior to the test and during the test. Before applying the standard spray, the insulator shall be given a thorough preliminary wetting either by immersion, by spraying in an inverted position or by using a hose on the insulator in its test position.

The test shall be performed by applying voltage between the rod fastened to the head and the steel pin, and raising it at a rate of approximately 10,000 v. every 15 sec. to a value at which wet flashover occurs. The initial applied voltage may be raised quickly to approximately 80 per cent of the wet flashover voltage.

A3. *Flashover Tolerances.*[1]—The flashover voltages of a pin-insulator design shall be considered as checked, if the average of the dry flashover voltages of three insulators shall be within 5 per cent and the average of the wet flashover voltages of three insulators shall be within 10 per cent of the corresponding assigned voltage.

A4. (*a*) *Puncture Test.*—Puncture test shall be performed with the insulator inverted and so immersed in insulating oil that the oil will be at least 6 in. (15.24 cm.) deep over all parts of the insulator. The insulating oil shall be capable of withstanding 15 kv. between 1 in. (2.54 cm.) disks spaced 0.1 in. (0.254 cm.) apart. The head of the insulator shall be covered with a cap of conducting cement approximately 1 in. (2.54 cm.) thick and 0.75 in. (1.90 cm.) larger in radius than the insulator head. The pin hole shall be provided with a cemented thimble in which shall be inserted a close fitting metal pin. Oil[2] may be excluded from the space between the pin and the thimble by means of cement.

The test shall be performed by applying voltage between the cement cap and the steel pin, and raising it at a rate of approximately 10,000 v. every 15 sec. to a value at which puncture occurs. The initial applied voltage may be raised quickly to the dry flashover voltage.

(*b*) Puncture test shall be made on insulators which have passed the routine flashover tests. Rejection of insulators for puncture shall be based on the "Per Cent Average Variation" in puncture voltage determined as described in Paragraph A5.

A5. *Determination of "Per Cent Average Variation" in Puncture Test.*—Purchaser will select from the insulators offered for final inspection not more than one-half of one per cent of the total quantity and not less than three insulators.

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[1] These flashover tolerances are made to provide for testing variables which are difficult to control.

[2] The procedure for testing oil is completely described in the Standard Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation: D 117) of the American Society for Testing Materials, 1936 Book of A.S.T.M. Standards, Part II, p. 1167.

Let
$V_1\ V_2\ V_3\ \ldots\ V_n$ = individual puncture values
V = average puncture voltage
$V = (V_1 + V_2 + V_3\ \ldots\ + V_n)/n$
Let
$a_1 = V - V_1 \qquad a_2 = V - V_2$
$a_3 = V - V_3 \qquad a_n = V - V_n$

Consider all the values of a as positive, that is, neglect the signs.

Let
a = average variation
A = per cent average variation
Then
$a = (a_1 + a_2 + a_3\ \ldots\ + a_n)/n$
$A = 100\,a/V$*

If "Per Cent Average Variation," A, exceeds 10 per cent, the entire quantity shall be rejected, or at the manufacturer's option and expense an additional 2 per cent or a minimum of 10 insulators may be tested. In this case the results of the second test alone shall be considered and if the "Per Cent Average Variation," A, exceeds 10 per cent the entire quantity represented shall be rejected. Further if the average puncture voltage V is less than $133\frac{1}{3}$ per cent of rated dry flashover voltage, the entire quantity represented shall be rejected.

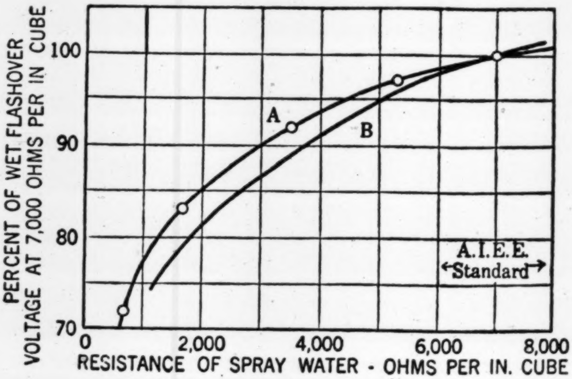

FIG. 2.

A6. *Variation of Wet Flashover Voltage with Water Resistance.*—It is frequently difficult to get consistent results for wet flashover voltages of insulators when the tests are made in different laboratories. This is sometimes largely due to the difference in resistance of the spray water. Tap water is sometimes 2000 ohms per inch cube or less, and steam condensate is sometimes over 50,000 ohms. In the Standards it is stated that the water shall have a resistance of from 6000 to 8000 ohms per inch cube, so that it always should be possible to obtain spray water of the specified resistance by treatment of the condensate or by mixing condensate with tap water.

The effect of using low-resistance water is well shown by the two curves given in Fig. 2, which are from independent sources. Curve A was contributed by Mr.

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* **_Example._—Five insulators punctured at: 150, 135, 145, 138, 142 kv., respectively:**

$V = (150 + 135 + 145 + 138 + 142)/5 = 142$
$a = (8 + 7 + 3 + 4 + 0)/5 = 4.4$
$A = 100 \times 4.4/142 = 3.09$ per cent

F. W. Peek, Jr., and Curve *B* was adapted from a discussion of the German Standards appearing in *Elektrotechnische Zeitschrift*. Mr. Peek's curve is based on tests of three, five and eight-unit suspension strings of 10-in. disks. The German curve is described as applying to "Cap type, insulators, flat shape; small suspension insulators; disk type strain insulators with metal skirt." In each case, 100 per cent flashover voltage is taken as the flashover voltage with water of 7000 ohms per in. cube resistance, this being half-way between the extremes permitted by the Standards.

A7. *Method of Reducing Dry Flashover Tests to Standard Humidity.*—For reducing dry flashover test voltages determined under conditions of humidity different from standard to standard humidity conditions the following method will be acceptable although it is expected that experience will result in the development of a simpler method.

Flashover voltages for a particular insulator design may be measured at three different humidities, at least one test being made at a humidity higher than standard and at least one test being made at a humidity lower than standard. The flashover voltage for standard humidity shall then be determined by graphical interpolation along a smooth curve drawn through the three points determined by plotting the test results with flashover voltage and vapor pressure as coordinates.

For the purpose of controlling humidity, a pin-type insulator and its support may be placed within an inclosure not less than 10 ft. (3.05 m.) cube. In the case of suspension insulators, the insulator or string and its support may be placed within an inclosure not less than three times the string length in any dimension, and in no case less than 10 ft. (3.05 m.) cube.

Insulators under test shall be at approximately the same temperature as the surrounding air.

TENTATIVE METHODS OF TEST

FOR

INSULATION RESISTANCE OF ELECTRICAL INSULATING MATERIALS[1]

A.S.T.M. Designation: D 257 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These methods cover the determination of insulation and volume resistance and approximate surface resistance of electrical insulating materials; also means of converting the latter two into terms of volume and surface resistivities.

DEFINITIONS

Definitions

2. (*a*) *Insulation Resistance.*—The insulation resistance between two electrodes which are fastened to or imbedded in a solid insulating material, or immersed in a liquid insulating material, is the ratio of the voltage applied to the electrodes to the total current which flows between the electrodes.

(*b*) *Volume Resistance.*—The volume resistance between two electrodes is the ratio of the voltage applied to the electrodes to the current which flows through the volume of the insulating material.

(*c*) *Volume Resistivity.*—The volume resistivity is the resistance between two electrodes which cover opposite faces of a centimeter cube provided the resistance of the surface layer is so high that it is not responsible for any appreciable part of the current.

(*d*) *Surface Resistance.*—The surface resistance between two electrodes is the ratio of the voltage applied to the electrodes to the current which flows through the surface layers.

(*e*) *Surface Resistivity.*—Surface resistivity is four times the resistance between two electrodes covering opposite faces of a cube when the volume resistance is so high that practically all of the current flows through the surface layers. This is equivalent to defining surface resistivity as the resistance between two opposite edges of a square of the surface layers.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

These methods are in effect a tentative revision of and will supersede, when adopted as standard, the Standard Methods of Test for Resistivity of Insulating Materials (A.S.T.M. Designation: D 257 - 33), 1936 Book of A.S.T.M. Standards, Part II, p. 1203.

NOTE 1 (Applicable particularly to solids).—In the definition of Insulation Resistance, the total current may consist of that which flows through a surface layer, that which flows through the volume of the material or, as is usually the case, a combination of the two. It is not practicable to make an accurate differentiation between surface leakage and volume leakage. The methods contained herein, however, permit of an approximation adequate for all practical purposes.

NOTE 2.—In the definitions of Insulation Resistance, Volume Resistance and Surface Resistance, the resistances are defined in terms of the current which is not a constant but depends on the time that the circuit has been closed. After the circuit is closed, the current often changes quite rapidly. For the purpose of tests conducted under these methods the current is measured at the end of 1 min.

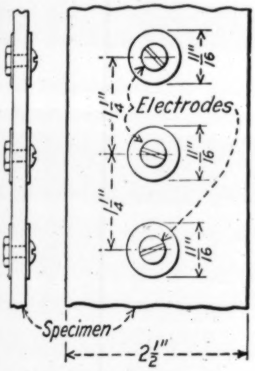
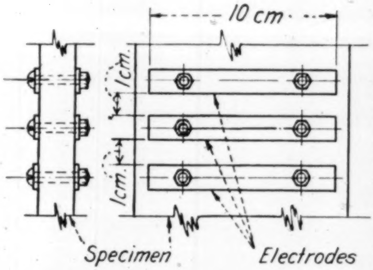


FIG. 1.—Application of Binding Post Electrodes to Flat, Solid Specimens.



Note - The Electrodes are either Tin Foil or other Conducting Material applied to Both Sides of the Specimen. In the Case of Tin Foil, it is wrapped around Metal Bars. After the Bars have been mounted in Position, the Tin Foil is pressed down with a thin Tool along the Edge of the Bar to assure Intimate Contact with the Insulation. The Suggested Dimensions have been found Convenient.

FIG. 2.—Application of Strip Electrodes to Flat, Solid Specimens.

TEST SPECIMENS AND TYPES OF ELECTRODES

A. *For Solid Insulating Materials*

Test Specimen

3. The test specimen for solid insulating materials shall be either in the form of a flat plate or a tube.

Electrodes

4. It is important that good contact be made between the electrode and the insulating material. For repeated tests at high relative humidities it is essential that the electrode metal be protected against tarnish. For insulation resistance determinations, the electrodes shown in Fig. 1 (binding posts), Fig. 2 (metal strips) or Fig. 3 (tapered pins) shall be used. For separating the insulation resistance into approximate surface resistance and volume resistance (from which approximate resistivities may be calculated) the guarded mercury electrodes (Fig. 4) are satisfactory though rather difficult to manipulate under abnormal atmospheric conditions.

Electrodes for Volume and Surface Resistance

5. When both the volume and surface resistances are to be measured, there shall be applied to each specimen three electrodes designated, respectively, as the unguarded electrode, the guarded electrode, and the guard electrode. In the case of a flat specimen, the guard electrode shall be in the form of a ring surrounding the guarded electrode as shown in Fig. 4.

NOTE.—In the case of a few non-absorbent materials such as hard rubber and glass the insulation resistance measurement may be considered a measurement of the surface resistance from which the surface resistivity may be calculated. Such assumptions are undesirable, however, because they invariably lead to generalization at the expense of accuracy. Consequently, the plain insulation resistance test should be used except for research purposes.

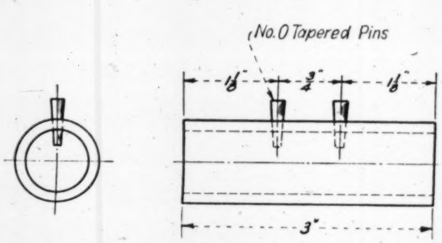


FIG. 3.—Application of Tapered Pin Electrodes to Solid Specimens.

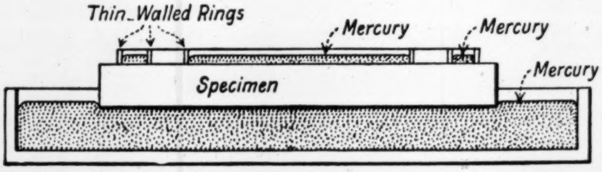


FIG. 4.—Application of Guarded Mercury Electrodes to Flat, Solid Specimens.

B. For Liquid Insulating Materials

Sampling

6. Samples of oil shall be obtained as described in Sections 2 to 7 of the Standard Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation: D 117) of the American Society for Testing Materials.[1] Samples of other liquid dielectrics shall be obtained in a similar manner with regard to cleanliness of containers and sampling apparatus and shall be representative of the lot to be tested. The quantity of the sample depends upon the type of resistivity cell used, but in any case it shall be sufficient to permit three separate resistivity determinations.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1167.

Electrodes

7. Suitable electrodes for liquid insulating material are parallel planes, concentric cylinders, or coaxial cones. The distance between the electrodes shall be not less than 0.75 mm. (0.03 in.) nor more than 5 mm. (0.2 in.). The voltage gradient shall not exceed 1200 v. per mm. (30 v. per mil). The area of the electrodes shall be sufficiently large so that the current flow can be measured, with the apparatus available, to an accuracy of 5 per cent.

NOTE 1.—Electrode areas of 50 to 500 sq. cm. (7.7 to 77.5 sq. in.) should prove suitable.

NOTE 2.—Because of the catalytic action of some metals on oils, the electrodes shall be plated with nickel, gold or platinum.

NOTE 3.—If the shape of the electrodes is such that their effective area and the distance between them is difficult to measure, the volume resistivity may be calculated by the use of the following formula:

$$K = 36\pi \times 10^{11} \times C$$

where K = the constant by which the measured resistance is to be multiplied to give the volume resistivity, and

C = the capacitance in farads of the electrode system with air as the dielectric. For methods of measurement of C, see the Tentative Methods of Testing Electrical Insulating Materials for Power Factor and Dielectric Constant (A.S.T.M. Designation: D 150 – 36 T) of the American Society for Testing Materials.[1]

APPARATUS

Apparatus

8. (*a*) The apparatus shall consist of a source of direct-current potential, a galvanometer with suitable shunts, a calibrating resistance, reversing switches and keys. The apparatus shall conform to the requirements specified in the following Paragraphs (*b*) to (*e*).

(*b*) *Direct-Current Potential.*—A dry or storage battery may be used as a source of potential. A rectified alternating-current voltage supply may also be employed, if precautions are observed to maintain a sufficiently constant direct-current voltage.

(*c*) *Galvanometer.*—The galvanometer shall have as high a sensitivity as is consistent with reasonable stability of zero. A sensitivity of 10^{-10} amp. per milliradian (1 mm. at 1-m. scale distance) is sufficient to measure resistance as high as 10^{11} ohms with an accuracy of ±10 per cent. It should be well damped. The most convenient shunt is the type known as a universal or Ayrton shunt, whereby that fraction of the total current which passes through the galvanometer may be changed without changing the galvanometer damping.

(*d*) *Calibrating Resistance.*—The calibrating resistance shall be at least 100,000 ohms. One megohm is preferable for ordinary measurements.

(*e*) *Switches and Keys.*—All switches and keys shall be suitably insulated and guarded.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 955 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1069.

NOTE 1.—If resistances higher than 10^{11} ohms are to be measured, a ballistic or a quadrant electrometer method is often employed. The latter method is described in the National Bureau of Standards *Scientific Paper No. 234.*[1] Other methods of measuring the minute currents flowing through these high resistances, employing vacuum tube electrometers,[2] are available and their use is permitted.

NOTE 2.—A bridge method of measuring insulation resistances up to 10^{11} ohms may be used, if a high-resistance balance detector, such as a vacuum-tube voltmeter, is employed, and the necessary guarding requirements are observed.

MEASUREMENTS

Measurements for Insulation Resistance

9. The following measurements shall be made when insulation resistance is to be determined:

(*a*) Dimensions of the electrodes (Figs. 1, 2 and 3),
(*b*) Distance between or spacing of the electrodes,
(*c*) The insulation resistance between the electrodes,
(*d*) The temperature of the material under test (see Section 11 (*a*)),
(*e*) The relative humidity of the surroundings (see Section 11 (*a*)), and
(*f*) Voltage used in measuring.

Measurements for Volume and Surface Resistance

10. The following measurements shall be made when both volume and surface resistance are to be determined by means of the specimen shown in Fig. 4:

(*a*) Area of the guarded electrode,

(*b*) Distance between the guarded electrode and the guard electrode,

(*c*) The over-all insulation resistance, that is, the resultant of the surface and volume resistances, when the measurement is made with the guard electrode connected to the unguarded electrode,

(*d*) The volume resistance, which is the resistance between the guarded electrode and the unguarded electrode when the guard electrode is maintained at substantially the same potential as the guarded electrode,

(*e*) The temperature of the material under test (see Section 11 (*a*)),

(*f*) The relative humidity of the surroundings (see Section 11 (*a*)),

(*g*) Voltage used in measuring, and

(*h*) Thickness of the specimen.

NOTE 1.—In the case of insulating oils it is often desirable to make measurements at an elevated temperature. To determine the suitability of oil for commercial apparatus tests are usually made at 85 C. or 100 C., or both.

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[1] H. L. Curtis, "Insulating Properties of Solid Dielectrics," National Bureau of Standards *Scientific Paper No. 234*, Vol. 11, p. 364.

[2] D. B. Penick, "Direct Current Amplifier Circuit for Use with the Electrometer Tube," *Review of Scientific Instruments*, Vol. VI, p. 113, April, 1935.

PROCEDURE

Conditioning

11. (*a*) Determinations of insulation resistance of solid dielectrics shall be made only on specimens which have been kept for at least 96 hr. in air at a definite humidity and temperature. The specimen shall be kept in air at this humidity during the determination. The measurements shall be made without opening the humidity chamber. Where electrode leads pass through the walls of the chamber, care shall be taken to insure against current leakage at these points by suitable guarding or by passing leads through bushings of wax or other materials of exceptionally high resistance.

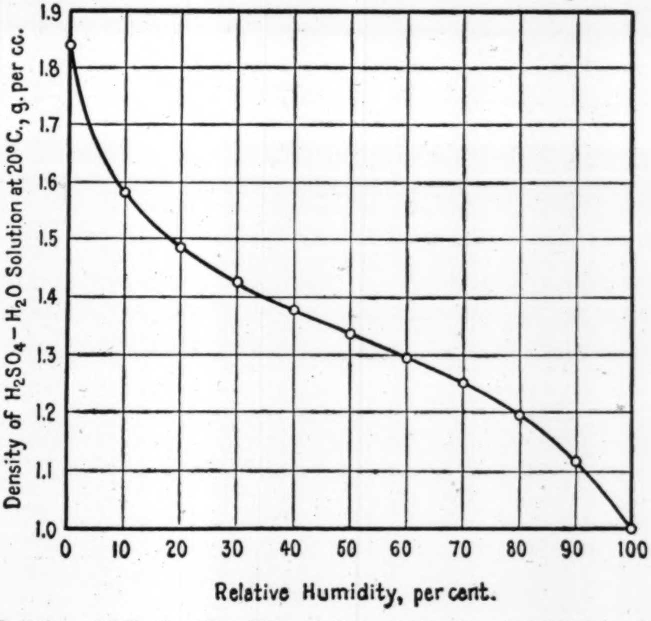

FIG. 5.—Relation of Relative Humidity of Air and Density of Acid-Water Mixture.

(*b*) *Humidity Control.*—The humidity may be readily maintained at any desired point by using a reasonably air-tight chamber in which a sulfuric acid - water mixture is exposed. The volume of the acid mixture shall be at least one-fiftieth of the volume of the chamber. The container for the mixture shall be an open vessel of such size that the area of the exposed liquid is at least three times as large as one side of a cube which would hold the required acid mixture. The density of the sulfuric acid - water mixture to give any desired humidity is shown by the curve in Fig. 5. No material which absorbs moisture, other than the test specimens, shall be allowed in the chamber. Provision shall be made for circulating the air and for maintaining the temperature constant. In case a lamp is placed inside the chamber, the specimens should be shielded from direct radiation. A desiccator may be used for a constant humidity chamber provided it can be kept where the temperature is constant. Saturated solutions of certain salts are also useful for maintaining constant humidity. For example, over a range of temperatures from 20 to 80 C. a saturated solution of sodium chloride maintains a humidity very close to 75 per cent relative humidity.

Method of Measuring Resistance

12. The resistance may be measured by means of a galvanometer used as an ammeter. The galvanometer shall have a suitable shunt for increasing its range. Figure 6 shows the diagram of connections and guarding of circuits, which are suitable for use in measuring the insulation resistance of specimens of the types shown in Figs. 1, 2 and 3, or for the approximate separation of surface and volume resistance by means of the arrangement shown in Fig. 4.

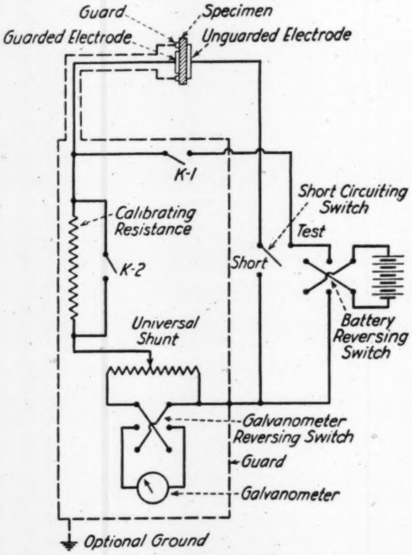
Guard
Specimen
Guarded Electrode
Unguarded Electrode
K-1
Calibrating Resistance
K-2
Short Circuiting Switch
Test
Short
Battery Reversing Switch
Universal Shunt
Galvanometer Reversing Switch
Guard
Galvanometer
Optional Ground

FIG. 6.—Diagram of Connections for Determinations of Insulation Resistance.

Insulation Resistance Determination

13. (*a*) The insulation resistance required in Section 10 (*c*) shall be determined as follows: Connect the leads to adjacent terminals on the specimen. Set the universal shunt at the position of lowest galvanometer sensitivity, close the short-circuiting switch to "Test" and the battery

reversing switch to the desired polarity. Increase the shunt ratio until a readable deflection is obtained on the galvanometer. Read the deflection 1 min. after closing the reversing switch. Open the reversing switch and close the short-circuiting switch to "Short."

(*b*) The resistances required in Section 10 (*c*) and (*d*) shall be determined by the deflection method, using the completely guarded circuit (Fig. 6) including the guard electrode on the specimen. The procedure is the same as that specified in Paragraph (*a*). In either case, if no appreciable deflection is obtained when the universal shunt is set at the position of maximum galvanometer sensitivity, the resistance is too high to be measured without resorting to one of the methods mentioned in Note 1 under Section 8.

Precautions

14. (*a*) *Mounting Specimens.*—In mounting the specimens for measurement, it is important that there shall be no conductive paths between the electrodes except those through the specimen. For example, the specimens shown in Figs. 1, 2 and 3 shall be held by one or both edges so that no one of the electrodes touches the supports.

(*b*) *Measuring High Resistances.*—In measuring high resistances by the set-up shown in Fig. 6, it is important that all parts of the apparatus between the guarded electrode and the battery terminal of the galvanometer shall be guarded, and adequately insulated from the guard. This applies also to key, K_1. The guard shall be sufficiently insulated from ground and from contact with the operator to prevent failure of the battery by leakage and danger of shock to the operator. Apparatus between the guarded electrode and the battery terminal of the galvanometer shall be sufficiently insulated from the guard so that the leakage shall not form an appreciable shunt on the galvanometer. This insulation resistance need be only 1 megohm.

(*c*) *Measuring Volume Resistance.*—In measuring the volume resistance by the deflection method it is important that the current flow between the guarded electrode and the guard electrode shall be negligible. The current through the specimen divides between this path and that through the calibrating resistance and galvanometer with its shunt, and only the flow through the galvanometer is measured. In order that the error resulting from this source shall be less than 1 per cent, the insulation resistance between the guarded electrode and the guard electrode shall be more than 100 times the combined resistance of the calibrating resistance and the galvanometer with its shunt. The key, K_2, which short circuits the calibrating resistance when closed, makes it possible to reduce this error by reducing the ratio of the resistance of the galvanometer path to that of the insulation resistance between guarded and guard electrodes by a factor of about 1000. If an appreciable change of deflection is seen when key K_2 is opened or closed, it is an indication that the leakage is large enough to be guarded against, and only the reading with key K_2 closed should be used in calculating the volume resistance.

(*d*) *Calibration.*—In calibrating, the specimen shall be short circuited by any convenient means, and with the same voltage applied that was applied to the specimen, the galvanometer deflection produced shall be read when only the calibration resistance is in series with it, using a suitable shunt ratio.

CALCULATIONS

Volume Resistance

15. If the calibrating resistance has a sufficiently high value, and a suitable universal shunt is provided, the resistance of a specimen may be easily compared with the calibrating resistance. The shunt setting may be represented by a fractional ratio, s, in negative powers of ten, or by a multiplying factor, f, in positive powers of ten. In the former case, when measuring the specimen, call the shunt ratio, S, and the galvanometer deflection, d. Let the galvanometer deflection observed during calibration (Section 14 (*d*)) be represented by, D, and the corresponding shunt ratio by, S; then if the calibrating resistance has a value, M, the resistance of the specimen, R, may be calculated from the following formula:

$$R = M \cdot \frac{D}{d} \cdot \frac{s}{S} - M \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Or, if the shunt setting is given in multiplying factors, the formula becomes:

$$R = M \cdot \frac{D}{d} \cdot \frac{F}{f} - M \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

where F = the shunt setting for the calibrating resistance, and
f = the shunt setting for the specimen.

If the calibrating resistance is so small that the battery will give too large a deflection of the galvanometer with the smallest shunt ratio (or largest multiplying factor), it is then necessary to calibrate by means of a low-voltage battery. The same measurements shall be made as in the previous cases, but it is now necessary to know the voltage of the calibrating battery, E, and of the measuring battery, e. Then:

$$R = M \cdot \frac{D}{d} \cdot \frac{s}{S} \cdot \frac{e}{E} - M \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

or

$$R = M \cdot \frac{D}{d} \cdot \frac{F}{f} \cdot \frac{e}{E} - M \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (4)$$

Insulation Resistance

16. The insulation resistance is the resistance as measured between any two adjacent binding posts of Fig. 1, between any two adjacent metal strips of Fig. 2, or between the tapered pins of Fig. 3. This value should be expressed merely as "insulation resistance." Where it is a certainty that the leakage is confined entirely to the surface, results so obtained may be converted to surface resistivity in terms of "surface resistance per square" by using the following relation for the disks:

$$\sigma = \frac{\pi R'}{L_n\left[\frac{d}{2r} + \sqrt{\left(\frac{d}{2r}\right)^2 - 1}\right]} \quad \text{..................} \quad (5)$$

where R' = twice the measured resistance,
d = the distance between the centers of the disks,
r = the radius of the disks, and
L_n = the natural logarithm.

If d has a value between $3r$ and $4r$ the following approximate formula may be used:

$$\sigma = \frac{\pi}{2} \cdot \frac{R'(d+r)}{d-r} \quad \text{..........................} \quad (6)$$

In the case of the metal strips the surface resistance per square may be calculated by the following formula:

$$\sigma = \frac{R'b}{L} \quad \text{..............................} \quad (7)$$

where R' = twice the surface resistance as measured,
b = the average length of the electrodes, and
L = the distance between the electrodes.

Volume Resistivity

17. The volume resistivity, ρ, shall be calculated from the following formula:

$$\rho = \frac{RA}{L} \quad \text{..............................} \quad (8)$$

where R = the volume resistance as measured with the specimen shown in Fig. 3 in accordance with Section 10 (d),
A = the area of the guarded electrode, and
L = the average thickness of the specimen.

Surface Resistance

18. (*a*) The surface resistance may be calculated from the measurements made with the specimen shown in Fig. 3 in accordance with Section 10 (*c*) and (*d*). This is by far the most reliable method. The formula for calculating the surface resistance is as follows:

$$R' = \frac{R_s\ R}{R - R} \qquad (9)$$

where R′ = the surface resistance,
R_s = the over-all resistance, Section 10 (*c*), and
R = the volume resistance, Section 10 (*d*).

(*b*) *Surface Resistivity.*—The surface resistivity shall be calculated from the surface resistance as follows:

$$\sigma = \frac{R'b}{L} \qquad (10)$$

where σ = the surface resistivity,
R' = the surface resistance,
b = the average circumference of the guarded electrode and of the inner edge of the guard electrode, and
L = the distance between the electrodes.

REPORT

Report

19. The report shall include the information indicated in the following Paragraphs (*a*), (*d*), (*e*), (*f*), (*g*) and (*h*) and may include the items in Paragraphs (*b*) and (*c*):

(*a*) The over-all insulation resistance in ohms,
(*b*) The volume resistivity in ohm-cm. units,
(*c*) The surface resistivity in ohms,
(*d*) The temperature in degrees Centigrade,
(*e*) The percentage relative humidity,
(*f*) Time of exposure to that humidity,
(*g*) The voltage used when other than the specified voltage, and
(*h*) The type of electrodes.

TENTATIVE METHODS OF TESTING SHEET AND PLATE MATERIALS USED IN ELECTRICAL INSULATION[1]

A.S.T.M. Designation: D 229 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1925; REVISED, 1926, 1928, 1930, 1931, 1932, 1934, 1935, 1936, 1937.[2]

Scope

1. These methods are intended to apply to stiff, flat sheet and plate materials, such as phenolic and other types of laminated sheets, vulcanized fiber, hard rubber, asbestos composition board, etc., to be used as electrical insulation.

Definition of Terms

2. In referring to the cutting of the specimens and the application of the load, the following definitions apply:

Flatwise.—Load applied to the flat side of the original sheet or plate.

Edgewise.—Load applied to the edge of the original sheet or plate.

Lengthwise.—In the direction of the length of the sheet.

Crosswise.—In the direction at right angles to the length of the sheet.

NOTE.—When the sheet has the same length and width, one dimension shall arbitrarily be designated as the length, and the other as the width.

TENSILE STRENGTH

Apparatus

3. Any standard type of testing machine may be used. The machine shall be accurate to within 1 per cent on the lowest load for which it is used. Jaws which tighten under load, such as wedge grip jaws, shall be used with the specimen properly aligned.

Specimens

4. Tension test specimens shall conform to the dimensions shown in Fig. 1. The surfaces of the specimen shall be smooth and free from scratches.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

[2] Latest revision accepted by Committee E-10 on Standards, August 26, 1937.

Procedure

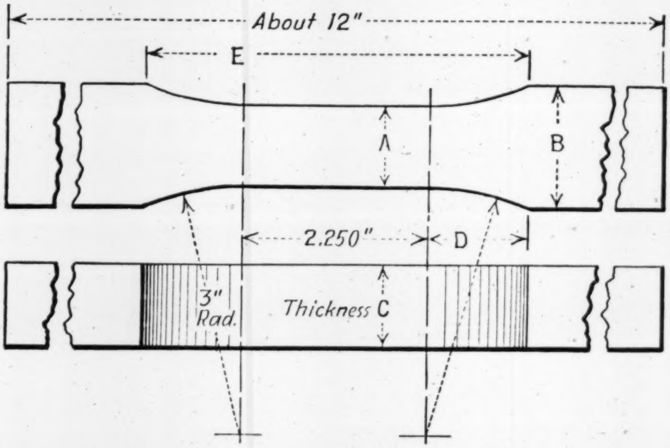
5. Five specimens cut lengthwise and five specimens cut crosswise of the sheet shall be tested.

Temperature

6. All tests shall be made at normal room temperature of about 20° C. (68° F.).

Speed of Testing Machine

7. The cross-head speed of the testing machine shall be such that the load can be accurately weighed, but shall not exceed 0.05 in. per minute when the machine is running idle.



When C is less than $\frac{1}{8}$ in., A is $\frac{1}{2}$ in., B is $\frac{3}{4}$ in., D is 0.857", E is 3.964 in.
When C is $\frac{1}{8}$ in. or over, A is 1 in., B is $1\frac{1}{2}$ in., D is 1.199", E is 4.648 in.

Fig. 1.—Tension Test Specimen for Laminated Sheet Insulating Materials.

Modulus of Elasticity

8. When it is desired to determine the elongation under load, a suitable extensometer measuring the elongation on a 2-in. gage length shall be used. The cross-head speed of the testing machine shall not exceed 0.03 in. per minute when the machine is running idle.

Procedure

9. The total change in length between zero load and a stress of 5000 lb. per sq. in. shall be determined.

Report

10. The report shall include the following:

(*a*) The direction in which the specimen was taken, whether lengthwise or crosswise of the sheet.

(*b*) The thickness and width of each specimen in inches expressed to the nearest 0.001 in. or in centimeters expressed to the nearest 0.025 mm.

(*c*) The breaking load of each specimen, in pounds or kilograms.

(*d*) The tensile strength of each specimen, in pounds per square inch or in kilograms per square centimeter.

(*e*) The location and nature of the fracture.

(*f*) The elongation in inches at a stress of 5000 lb. per sq. in., if determined.

(*g*) The room temperature.

FLEXURAL STRENGTH

(FORMERLY CALLED TRANSVERSE STRENGTH)

(*A*) *For Materials $\frac{1}{32}$ in. or Over in Thickness*[1]

Apparatus

11. (*a*) Any standard type of testing machine may be used. The machine shall have an accuracy of at least 1 per cent of the lowest load for which it is used.

(*b*) The specimen shall be tested as a simple beam loaded at the center. The supports shall have contact edges rounded to a radius of $\frac{1}{8}$ in. for material $\frac{1}{8}$ in. or over in thickness, and to a radius of $\frac{1}{16}$ in. for thinner materials. For material thinner than $\frac{1}{8}$ in. both supports shall be provided with a pin or other stop to align one side of the specimen perpendicular to the line of contact with the supports. The distance between the points of support shall be 4 in. for edgewise tests of all thicknesses and eight times the nominal thickness of the material to be tested for tests in the flatwise direction, except that the minimum span shall be $\frac{1}{2}$ in.

Specimen

12. The test specimen shall be $\frac{1}{2}$ in. in width except for specimens over $\frac{1}{2}$ in. in thickness tested in the flatwise direction, which shall have the width equal to the thickness of the specimen. The thickness shall be the full thickness of the sheet. The length shall be 1 in. greater than the distance between the points of support as specified in Section 11 (*b*).

Procedure

13. The specimens shall be tested as follows:

(*a*) Five specimens cut lengthwise of sheet, tested flatwise.

(*b*) Five specimens cut lengthwise of sheet, tested edgewise.

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[1] Conventional flexure tests in a flatwise direction are not recommended for materials thinner than $\frac{1}{32}$ in. nor in the edgewise direction for materials thinner than $\frac{1}{4}$ in.

(*c*) Five specimens cut crosswise of sheet, tested flatwise.

(*d*) Five specimens cut crosswise of sheet, tested edgewise.

Temperature of Tests

14. All tests shall be made at normal room temperature of about 20° C. (68° F.).

Speed of Testing Machine

15. The cross-head speed of the testing machine shall be such that the beam of the machine can be kept balanced, but shall not exceed 0.05 in. per minute when the machine is running idle.

Report

16. The report shall include the following:

(*a*) The directions of cutting and loading of the specimen as specified in Section 13.

(*b*) The thickness and width of each specimen in inches expressed to the nearest 0.001 in. or in centimeters expressed to the nearest 0.025 mm.

(*c*) The breaking load of each specimen in pounds or kilograms.

(*d*) The maximum fiber stress in pounds per square inch or kilograms per square centimeter calculated from the formula:

$$S = \frac{3\ Pl}{2\ bd^2}$$

where S = maximum fiber stress;
P = breaking load in pounds or kilograms;
l = distance between supports in inches or centimeters;
b = width of specimen in inches or centimeters;
d = depth of specimen in inches or centimeters.

(*e*) The room temperature.

COMPRESSIVE STRENGTH

Apparatus

17. Any standard testing machine may be used provided it is accurate within 1 per cent of the lowest load for which it is used. One end of the specimen shall bear upon an accurately centered spherical bearing block, located whenever practicable, at the top, and the metal bearing plates shall be directly in contact with the ends of the test specimen.

Specimen

18. (*a*) For sheets 1 in. in thickness or over, the test specimen shall be a 1-in. cube, the faces of which shall be flat and perpendicular to the axes, being ground to this condition if necessary.

(*b*) For sheets less than 1 in. in thickness, the specimen shall consist of a pile of sheets 1 in. square with a minimum number of layers to produce a height of at least 1 in.

Procedure

19. (*a*) For sheets 1 in. in thickness or over, three specimens shall be crushed in the condition in which they are received with the load applied flatwise, three specimens with the load applied edgewise-lengthwise of the sheet and three specimens with the load applied edgewise-crosswise of the sheet.

(*b*) For sheets less than 1 in. in thickness, three specimens shall be crushed in the condition in which they are received with the load applied flatwise of the sheet.

20. The load shall be applied on the ends of the specimen and the crosshead speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. (1.27 mm.) per minute when the machine is running idle. For the best results use the slowest possible speed.

Report

21. The report shall include the following:

(*a*) The dimensions of each specimen in inches expressed to the nearest 0.001 in. or in centimeters expressed to the nearest 0.025 mm.

(*b*) The direction of application of the load;

(*c*) The load on each specimen in pounds or kilograms at the first sign of failure;

(*d*) The ultimate compressive strength in pounds per square inch or kilograms per square millimeter, of each specimen, calculated from the area of each specimen measured before the load is applied;

(*e*) The general character of the material tested; with a description of how it acts under the applied load.

RESISTANCE TO IMPACT

Impact Test

22. (*a*) Resistance to impact shall be determined in accordance with the Tentative Methods of Testing Electrical Insulating Materials for Resistance to Impact (A.S.T.M. Designation: D 256 - 34 T) of the American Society for Testing Materials.[1]

Specimen

(*b*) Specimens for the Charpy test may be notched or unnotched, as desired, according to conditions of service, and for the Izod test shall be notched (Note 1). Specimens shall be cut both lengthwise and crosswise of the sheet (Note 2).

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 995 (1934); also 1937 Book of A.S.T.M. Tentative Standards, p. 1023.

NOTE. 1.—The unnotched specimen gives a higher strength than the notched specimen and the ratio of the strength of the unnotched specimen to that of the notched specimen varies with different materials.

NOTE 2.—Conditioning of the specimens of vulcanized fiber is necessary in order to secure consistent results. The procedure for conditioning is under consideration by the committee. In order to secure comparative results, the specimens should be conditioned at the same temperature and humidity.

WATER ABSORPTION TESTS

Scope

23. The water absorption tests are intended to determine the rate at which water is absorbed by the material when immersed, the total quantity absorbed at saturation, and the volatile content as received.

NOTE.—The electrical properties of different materials are not necessarily affected to the same extent by the same increase in moisture content so that the water absorption test must be correlated with the desired electrical tests.

The water immersion test and exposure to air of high humidity are not always directly comparable and should be considered before substituting one for the other.

(*A*) *Rate of Absorption*

Specimen

24. The test specimen shall be 3 by 1 in. by the thickness of the material. It shall be sawed or sheared from the sample so as to have smooth edges free from cracks. The cut edges of all test specimens shall be finished with No. 0 or finer sandpaper or emery cloth. Sawing and sandpapering operations should be slow enough so that the material is not heated appreciably. The thickness in inches to the nearest 0.001 in. shall be measured on the original specimen at the center.

Procedure

25. To determine the rate of absorption, two specimens shall be tested individually as follows:

(*a*) The specimen shall be weighed, dried in an oven for 1 hour at from 105 to 110° C., cooled in a desiccator and immediately re-weighed. The difference between the original weight and the dry weight shall be recorded as loss in weight on drying. The percentage loss in weight shall be calculated on the original weight.

(*b*) The dried specimen shall then be immersed in distilled water maintained at a temperature of 25° C. ± 2° C. (77° F. ± 3.6° F.) throughout the test. At the end of 2 hours the specimen shall be removed from the water, the surface water wiped off with a dry cloth and the specimen weighed immediately and then replaced in the water.

If the specimen is $\frac{1}{16}$ in. in thickness or less it shall be weighed in a weighing bottle. The difference between the weight of the wet specimen and that of the dry specimen shall be recorded as the weight of water absorbed in 2 hours. The percentage of water absorbed shall be calculated on the weight of the dry specimen.

(*c*) After 24 hours immersion the specimen shall again be removed from the water, the surface water wiped off with a dry cloth and the specimen weighed. The difference between the weight of the specimen after 24 hours immersion and the weight of the dry specimen shall be recorded as the water absorbed in 24 hours. The percentage of water absorbed shall be calculated on the weight of the dry specimen. If the individual 24-hour absorptions differ by more than 10 per cent from the average absorption of the two specimens the test shall be repeated.

Report

26. The report shall include the values for each of the two specimens and the average values as follows:

(*a*) The thickness of the specimen before the test, to the nearest 0.001 in.;

(*b*) The percentage loss in weight on drying calculated on the original weight;

(*c*) The percentage of water absorbed in 2 hours and in 24 hours calculated on the dry weight.

(*B*) *Total Absorption at Saturation*

Specimen

27. (*a*) The test specimen shall be 3 by 1 in. by the thickness of the material. It shall be sawed or sheared from the sample so as to have smooth edges free from cracks. The cut edges of all test specimens shall be finished with No. 0 or finer sandpaper or emery cloth. Sawing and sandpapering operations should be slow enough so that the material is not heated appreciably. The thickness in inches measured to the nearest 0.001 in. shall be measured on the original specimen at the center and on the reduced specimen in case a reduced specimen is used (see Paragraph (*b*)).

(*b*) When the sample for determining total absorption is so thick that too long a time is required for saturation using the full thickness, the thickness may be reduced as follows: The sample shall be machined to a thickness of about $\frac{5}{64}$ in., both surfaces parallel to the laminations being machined off. This may readily be done with a smooth-cut circular saw. It shall then be sanded to $\frac{1}{16}$ in. in thickness, finishing with No. 0 or finer sandpaper. While the sanded surfaces of the sample should be approximately parallel with the laminations, the thickness may vary ±0.010 in., since the object is to provide comparable specimens which will reach saturation in a reasonable time. The machined and sanded sample shall then be cut into specimens 3 by 1 in. and the edges sanded.

Procedure

28. To determine the total water absorption at saturation, two specimens shall be tested individually as follows:

(*a*) The specimen shall be weighed, dried in an oven for 1 hour at from 105 to 110° C., cooled in a desiccator and immediately reweighed. The difference between the original weight and the dry weight shall be recorded as loss of weight on drying. The percentage loss in weight shall be calculated on the original weight.

(*b*) The dried specimen shall then be immersed in distilled water at a temperature of from 20 to 30° C. At the end of 24 hours the specimen shall be removed from the water, the surface water wiped off with a dry cloth and the specimen weighed immediately and then replaced in the water. The weighings shall be repeated every day for the first week and every week thereafter until the increase in weight, as shown by three consecutive weighings, averages less than 1 per cent of the total increase in weight, when the specimen shall be considered to be saturated.

(*c*) The difference between the saturated weight and that of the dry weight shall be considered as the water absorbed at saturation. The percentage of water absorbed shall be calculated on the dry weight of the specimen.

Report

29. This report shall include the values for each of the two specimens as follows:

(*a*) The original thickness of material, measured to the nearest 0.001 in.;

(*b*) If a reduced specimen is used, the thickness measured to the nearest 0.001 in.;

(*c*) The percentage loss in weight on drying, calculated on the original weight;

(*d*) The percentage of water absorbed at saturation, calculated on the dry weight.

(C) *Volatile Matter*

Specimen

30. The test specimen shall conform to the requirements specified in Section 12.

Procedure

31. To determine the volatile matter, two specimens shall be tested individually as follows:

The specimen shall be weighed, dried in an oven at from 105 to 110° C. until the decrease in weight as shown by two consecutive weighings at least 24 hours apart is less than 1 per cent of the total decrease in weight. The difference between the original weight and the dry weight shall be recorded as the volatile matter present in the specimen as received (this includes in addition to water any volatile solvent left during manufacture). The percentage of volatile matter shall be based on the weight of the dry specimen.

Report

32. The report shall include the values for each of the two specimens as follows:

(*a*) The original thickness of the material measured to the nearest 0.001 in.;

(*b*) The percentage of volatile matter, based on the weight of the dry specimen.

DIELECTRIC STRENGTH

Dielectric Strength

33. The dielectric strength shall be determined in accordance with the Tentative Methods of Testing Sheet, Tape, and Molded Insulating Materials for Dielectric Strength (A.S.T.M. Designation: D 149 – 36 T) of the American Society for Testing Materials.[1]

PHASE DIFFERENCE (POWER FACTOR) AND DIELECTRIC CONSTANT

Dielectric Constant

34. The phase difference and dielectric constant shall be determined in accordance with the Tentative Methods of Testing Electrical Insulating Materials for Power Factor and Dielectric Constant (A.S.T.M. Designation: D 150 – 36 T) of the American Society for Testing Materials.[2]

BONDING STRENGTH

For Laminated Materials $\frac{3}{16}$ in. or Over in Thickness

Apparatus

35. Any standard type of testing machine may be used, providing it is accurate within 1 per cent of the lowest load for which it is used. To this shall be fitted a head containing a 10-mm. steel ball.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 918 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1002.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 955 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1069.

Specimens

36. Test specimens shall be 1 in. square, smoothly and accurately cut on a smooth saw.

Procedure

37. Five specimens shall be tested. These shall be placed on edge and the steel ball shall center accurately between the edges and ends of the specimen.

Temperature

38. All tests shall be made at normal room temperature of about 20° C. (68° F.).

Speed of Testing Machine

39. The speed of the cross-head of the testing machine shall be such that the load can be accurately weighed, but shall not exceed 0.05 in. per minute when the machine is running idle.

Report

40. The report shall include the following:

(*a*) the thickness of the material; and

(*b*) the load, expressed in pounds or kilograms, required to split the specimen.

ROCKWELL HARDNESS

Apparatus

41. (*a*) The Rockwell hardness tester shall be used, conforming to the requirements of the Standard Methods of Rockwell Hardness Testing of Metallic Materials (A.S.T.M. Designation: E 18) of the American Society for Testing Materials.[1]

(*b*) The readings shall be taken on the red scale and shall be recorded as follows: Count the number of times the needle passes through zero on the red scale on the application of the major load. Subtract from this the number of times the needle passes through zero upon the removal of this load. If the difference is zero, the reading shall be recorded as over 100. If the difference is 1, the reading shall be recorded as between 0 and 100 and, if the difference is 2, the reading shall be recorded as negative.

Specimen

42. (*a*) The specimen shall have a minimum thickness of $\frac{1}{4}$ in. The specimen may be composed of a pile-up of several pieces of the material of the same thickness, providing that precaution is taken that the surfaces of the pieces are in contact and not held apart by burrs from saw cuts, etc. The area of the specimen shall be 1 in. square if cut from sheet stock or other shape of at least this area.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 824.

(*b*) Care shall be taken that the test is not made so near the edge of the specimen that it will break out when the major load is applied.

(*c*) The specimen shall be supported in such a manner as to prevent errors due to overhang, when it does not balance itself on the anvil.

(*d*) All tests shall be made at a temperature of 25 C. ± 2 C. (77 F. ± 3.6 F.).

NOTE 1.—It should be noted that some materials such as laminated phenolic plates show an appreciable variation in the Rockwell hardness with variation in temperature as small as 8° C. (15° F.).

NOTE 2.—Conditioning of the specimens of vulcanized fiber is necessary in order to secure consistent results. The procedure for conditioning is under consideration by the committee. In order to secure comparative results, the specimens should be conditioned at the same temperature and humidity.

Procedure

43. (*a*) The procedure outlined in the Standard Methods E 18[1] shall be followed. The adjustment of speed of load application and the time of application of the major load are of great importance. The machine shall be so adjusted that the major load will be fully applied in from 6 to 8 seconds as a longer time will give an erroneous result due to "creeping" of some materials. It is important that the major load be released immediately after it has been fully applied.

(*b*) For phenolic laminated sheets, a $\frac{1}{4}$-in. ball penetrator, a minor load of 10 kg. and a major load of 100 kg. shall be used in connection with the red scale. This combination is known as the M scale.

(*c*) For vulcanized fiber, a $\frac{1}{2}$-in. ball penetrator, a minor load of 10 kg. and a major load of 60 kg. shall be used. This combination is known as the R scale. The major load shall be removed within seven to eight seconds after the handle has been tripped.

Report

44. The report shall include the following:

(*a*) The Rockwell hardness number read on the red scale, the major load and the diameter of the penetrator. The letter M shall precede the Rockwell hardness number for phenolic laminated sheets and the letter R shall precede that for vulcanized fiber.

(*b*) The total thickness of the specimen;

(*c*) The number of pieces in a specimen and their average thickness, and

(*d*) The room temperature.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 824.

DENSITY (VULCANIZED FIBER)

Density

45. (*a*) *Fiber 0.020 in. or less in Thickness.*—For vulcanized fiber 0.020 in. or less in thickness the specimen shall be dried in an oven for 2 hr. at 105 to 110 C. It shall then be weighed on a chemical balance accurate to 0.25 per cent. The thickness shall be determined after drying. The apparent density shall be calculated from the thickness and the weight by means of the following formula:

$$\begin{array}{c}\text{Apparent Density,}\\ \text{g. per cu. cm.}\end{array} = \frac{\text{Weight g. per sq. cm.}}{\text{Thickness, in.}} \times 0.394$$

(*b*) *Fiber over 0.020 in. in Thickness.*—The density of vulcanized fiber over 0.020 in. in thickness shall be determined with any suitable weight-difference apparatus for making readings in water and air using the specimen as received.

ASH (VULCANIZED FIBER)

Specimen

46. The test specimen shall consist of 2 to 5 g. of finely divided particles, such as millings or filings, of the vulcanized fiber.

Procedure

47. The test specimen shall be dried for 2 hr. at 105 C., weighed, then ignited to constant weight in a crucible and weighed. The percentage of ash shall be calculated, based on the weight of the dried specimen.

ZINC CHLORIDE (VULCANIZED FIBER)

Specimen

48. The test specimen shall consist of 5 g. of finely divided particles, such as millings or filings, of the vulcanized fiber.

Procedure

49. (*a*) The test specimen shall be dried at 105 to 110 C. to constant weight or until two successive 30-min. weighings show a loss not greater than 0.2 per cent of the weight of the specimen.

(*b*) The dried specimen shall be placed in a Soxhlet, Wiley-Richardson, or similar type of extractor and extracted with distilled water for 3 hr. The rate of extraction with the Wiley-Richardson size of extractor must be sufficient to cause the tube containing the sample to be filled by condensation and emptied by the siphon at least six times per hour. When the Soxhlet or similar size extractor is used the tube must be filled and emptied at a rate of at least 3 cycles per hour. After this extract has cooled, 0.5 ml. of potassium chromate solution (10 per cent) shall be added and the extract titrated with standard silver nitrate solution. The end point is indicated by the appearance of a permanent red color. A blank test shall be made on an equal volume of distilled water and potassium chromate solution and the result shall be subtracted from that of the specimen. The net result shall be calculated in terms of zinc chloride.

SILICA (VULCANIZED FIBER)

Specimen

50. The test specimen shall consist of 5 g. of finely divided particles, such as millings or filings, of the vulcanized fiber.

Procedure

51. (*a*) The test specimen shall be dried at 105 to 110 C. to constant weight or until two successive 30-min. weighings show a loss not greater than 0.2 per cent of the weight of the specimen.

(*b*) The dried specimen shall be transferred to a crucible and slowly ignited to constant weight. The ash shall be wet with distilled water and transferred to a heat-resistant glass beaker. About 75 ml. of HCl (sp. gr. 1.19) shall be added and the beaker covered with a watch glass. The contents of the beaker shall be evaporated to dryness. To the residue shall be slowly added 10 ml. of HCl (sp. gr. 1.19) followed by 75 ml. of distilled water. The mixture shall be filtered through ashless filter paper and washed with cold water, then with warm water until the filtrate is free from chlorides. The filter paper shall be ignited to constant weight in a weighed platinum crucible. Then, 4 ml. of HF (48 to 60 per cent) shall be added and heat applied until all white fumes are driven off. The crucible shall be cooled and weighed. The difference between this weight and the previous one indicates silicon present as silica. The results shall be expressed as a percentage of the weight of the dried specimen.

FLAMMABILITY

Scope

52. This test is intended to evaluate the tendency of the material to burst into flame under the continuous application of heat.

Apparatus

53. The apparatus shall consist of the following:

(*a*) *Shielding Box.*—A shielding box, made either of metal or wood, in which the test is conducted for the purpose of warding off air currents. This box shall be 12 by 24 in. by 16 in. in height and shall contain no front or top.

(*b*) *Resistance Wire.*—Seven feet of No. 30 B. & S. gage nichrome wire having a resistance of 6.75 ohms per foot ±5 per cent.

(*c*) An electrical connection for alternating or direct current of 110 v. Where necessary, the voltage may be brought to this value with a rheostat.

(*d*) A stop watch, voltmeter, and stand clamp.

Specimen

54. (*a*) The test specimen shall be smooth-sawed from sheets to dimensions of $\frac{1}{8}$ in. ±0.005 in. by 1 in. ±0.005 in. by 8 in. The edges of the specimen shall be notched by a milling machine. The notches shall be rectangular, $\frac{1}{32}$ in. in depth and 0.015 in. in width, and shall be spaced 10 notches to the inch. Binding posts shall be fastened at each end of the specimen. The resistance wire shall be wound firmly about the specimen into the notches, and the ends shall be fastened to the binding posts.

(*b*) The test specimens shall be conditioned for 24 hr. at 105 C. before test.

(*c*) Five specimens shall be tested.

Procedure

55. The shielding box shall be placed within a hood. The specimens shall be mounted on the stand clamp in a horizontal position with the laminations in a vertical position inside the box. The front of the hood shall be lowered to within 2 in. of the bottom. The starting switch shall be snapped on and at that instant the stop watch started. The current shall be adjusted to the specified voltage and when the sample bursts into flame the watch shall be stopped and the time interval from the beginning of the test shall be recorded.

Report

56. The report shall state the number of seconds required for the specimen to burst into flame.

TENTATIVE METHODS OF TESTING SHELLAC USED FOR ELECTRICAL INSULATION[1]

A.S.T.M. Designation: D 411 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1935; Revised, 1937.

Scope

1. These methods cover tests for shellac in the dry or powdered form to be used for electrical insulating purposes.

Sampling and Chemical Tests

General Tests

2. Each of the following tests shall be made in accordance with the methods described in the Standard Methods of Sampling and Testing Shellac (A.S.T.M. Designation: D 29) of the American Society for Testing Materials:[2]

(*a*) Sampling.
(*b*) Insoluble Matter.
(*c*) Iodine Number.
(*d*) Moisture Content.
(*e*) Wax (Method *B*).
(*f*) Ash.

Polymerization Time

Apparatus

3. The apparatus required for the test for polymerization time shall consist of the following:

(*a*) *Test Tubes.*—Two $\frac{3}{4}$-in. glass[3] test tubes, supported by a wire rack to a depth of 4 in. when placed in an oil bath. It is essential that the test tubes be supported and maintained in a vertical position. The rack shall be so constructed as to permit free circulation of oil around the test tubes.

(*b*) *Oil Bath.*—An oil bath of such construction as to permit of maintaining a uniform test temperature within 1 C. It is essential to use a mechanical stirring device in the oil bath to maintain a uniformly distributed temperature. The oil used shall be a mineral oil having a Saybolt Universal viscosity of approximately 150 sec. at 100 C.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 760.

[3] Pyrex or quartz glass is satisfactory for this purpose.

(*c*) *Burner.*—A bunsen burner for heating the oil bath.

(*d*) *Glass Rod.*—A smooth glass rod 5 mm. in diameter. A small mark or indicator shall be placed on top of the rod or the glass formed-over to facilitate readings as outlined in Section 6 (*d*). The sides of the glass rod, near the end which is immersed in the shellac, shall be flattened slightly.

(*e*) *Thermometer.*—A suitable thermometer to indicate the test temperature.

Conditioning

4. (*a*) Samples of shellac before testing shall be rolled and mixed well on clean paper and then carefully dried as described in Paragraph (*b*).

(*b*) A flat-bottomed dish, about 4 in. in diameter containing a 5-g. sample of shellac, shall be placed in a well-ventilated gas or electric oven at a temperature of 41 C. ± 2 C. for approximately 16 hr. (or overnight). It is essential to have the sample spread out on the dish during the drying period.

(*c*) After drying, the sample shall be transferred immediately to a clean, dry bottle, and stoppered tightly, allowing it to cool in the bottle. The bottle shall not be opened except when a specimen is being removed for test.

Specimen

5. (*a*) Each specimen shall consist of 2 g. of the shellac taken from the stoppered bottle.

(*b*) Two specimens of each shellac sample shall be tested.

Procedure

6. (*a*) A specimen of shellac shall be transferred from the stoppered bottle to the $\frac{3}{4}$-in. glass test tube. The tube shall then be inserted and held securely in a vertical position in the test rack placed in the oil bath maintained at the specified test temperature. The temperature shall be maintained throughout the test within ±1 C. of the desired temperature.

NOTE.—The method is suitable for testing at any temperature between 125 and 225 C.

(*b*) The time when the test tube enters the oil bath shall be recorded.

(*c*) Using the 5-mm. glass rod, the specimen shall be stirred gently for the first 3 min. of the test so that the shellac becomes well melted in as short a time as possible. It is essential that there be no stirring after the first 3-min. period.

(*d*) At the end of each minute thereafter the glass rod shall be given a slight twist (turning approximately 90 deg.). In the early stages of the test, and before the polymerization point is reached, the glass rod will remain in the position to which it has been turned. When, however, the shellac takes on a rubbery set, there will be a definite turning or twisting back of the rod following the 90-deg. twist. This is the end point. (In the early stages of the test there may be a tendency for the glass rod to move back slowly after twisting with the fingers. This slight movement should not be confused with the definite "twist-back" observed when the end point is reached.)

(*e*) The temperature shall be recorded each minute during the test. The average of all these readings shall be considered the test temperature.

Polymerization Time

7. The elapsed time in minutes, from the time cf entry into the bath and including the 3-min. stirring period, until the first "twist-back" of the glass rod is noted, shall be recorded as the "polymerization time."

Report

8. The report shall include the following:

(*a*) The polymerization time to the nearest minute for each specimen.

(*b*) The average of the values in Paragraph (*a*).

(*c*) The test temperature.

FLOW TEST

Scope

9. (*a*) This method is intended to determine the flow of shellac when subjected to a definite temperature under the conditions of test indicated.

(*b*) The method consists in melting a sample of ground shellac in a graduated test tube and then tilting the tube to a definite angle while maintained at a stated temperature in order to permit the shellac to flow down the tube.

(*c*) The test provides for two methods, as follows:

Method A.—In this method the time required for the shellac to flow specified distances along the test tube is observed.

Method B.—In this method the total distance the shellac flows along the test tube in a specified time is observed.

Apparatus

10. (*a*) *Method A*.—A suitable form of testing apparatus for use in Method *A* is shown and described in the Appendix.

(*b*) *Method B*.—A suitable form of testing apparatus for use in Method *B* is shown and described in the Appendix. Any apparatus that will provide for accurately maintaining the required test temperature and the required positions of the test tubes may be used in Method *B*.

Specimen

11. (*a*) Each test specimen shall consist of 2 g. of shellac, coarsely ground (approximately 20-mesh). As changing atmospheric conditions

have a very marked effect on the flow of shellac, the open test tubes containing the test specimens shall be exposed to the laboratory atmosphere for not less than 24 hr. before testing.

(*b*) Two specimens shall be tested.

Preparation of Apparatus

12. (*a*) The two specimens of shellac shall be placed in separate glass test tubes, care being taken that the specimen in each tube is at the bottom and that none of the powdered shellac adheres to the walls of the glass tube. The top level of the dry shellac in each tube shall be read on the millimeter graduated scale. The tubes containing the specimens shall then be clamped in place in the testing fixture (see Section 10; also Fig. A1 in the Appendix).

(*b*) The testing fixture with the glass test tubes in a vertical position shall be inserted in an oil bath maintained at the desired test temperature to within ±1 C.

NOTE.—The method is suitable for testing at any temperature between 100 and 125 C.

(*c*) The top level of the specimen in each tube should be level and at right angles to the walls of the tube.

(*d*) The specimens shall be allowed to melt for 3 min.

Method A

Procedure, Method A

13. At the end of the 3-min. melting period, the test tubes shall each be placed at an angle of 15 deg. from the horizontal with the corked ends down (see Section 10 (*a*); also Fig. A2 in the Appendix) and with the breather tube extending above the level of the oil bath. The change from the vertical position to the flow position shall be made as quickly as possible. With the oil bath maintained at the desired test temperature, the total time required for the shellac in each tube to flow from the initial level of the shellac to each centimeter marking along the tube shall be recorded. The test in each tube shall be discontinued when the flow is 9 cm. or the total time is 20 min.

Report, Method A

14. The report of tests by Method *A* shall include the following:

(*a*) The time required for each centimeter distance of flow for each specimen,

(*b*) A curve showing the data reported in Paragraph (*a*) with time plotted as abscissa and flow in centimeters as ordinates,

(*c*) The test temperature,

(*d*) The angle of the test tubes during the flowing period, and

(*e*) The atmospheric temperature and humidity of the laboratory.

Method B

Procedure, Method B

15. (*a*) At the end of the 3-min. melting period, the test tubes shall each be placed at an angle of 15 deg. from the horizontal with the corked ends down (see Section 10 (*b*); also Fig. A2 in the Appendix) and if a breather tube is used, it shall extend above the level of the oil bath. The change from the vertical position to the flow position shall be made as quickly as possible. With the oil bath maintained at the desired test temperature, the test tubes shall be allowed to remain in the bath in this position for exactly 12 min.

(*b*) The test tubes shall then immediately be removed, placed in a vertical position, cooled, wiped, and the flow of the shellac in each tube measured by reading the distance between the initial point (Paragraph (*a*)) and the end of the flow tongue. The "feather" at the very tip of the tongue should be disregarded.

NOTE.—This "feather," caused by separation of wax from the shellac, is more noticeable with some shellacs than with others. It can readily be distinguished from the main body of shellac as it is always of different color.

Report, Method B

16. The report of tests by Method *B* shall include the following:

(*a*) The flow expressed in millimeters for each specimen,
(*b*) The average of the values in Paragraph (*a*),
(*c*) The test temperature,
(*d*) The angle of the test tube during the flowing period, and
(*e*) The atmospheric temperature and humidity of the laboratory.

APPENDIX

DESCRIPTION OF APPARATUS FOR DETERMINING THE FLOW OF SHELLAC

The apparatus shown in Figs. A1 and A2 consist of the following:

(*a*) *Test Tubes.*—Two test tubes, *A*, preferably of a heat-resistant glass, 5 in. in length, 1 in. in outside diameter, $\frac{1}{16}$ in. in wall thickness and graduated in millimeters for holding the shellac specimen, *S*. The test tubes are stoppered with tightly fitting corks through which are extended small glass breather tubes, *B*.

(*b*) *Support.*—A fixture, *C*, for holding the glass tubes in the correct position. This consists of two disks of brass supported as a pendulum as indicated. The disks are free to turn on the supporting shaft and are held in the desired position by the pin, *D*. The tubes are supported on narrow V-blocks, and held in position by coil springs, *H*, attached to disks, *C*.

(*c*) *Oil Bath.*—An oil bath, *E*, heated by a bunsen burner. Oil having a Saybolt Universal viscosity of approximately 150 sec. at 100 C. is suitable. Glycerine or a clear oil should be used where visual observations during the flowing period are required as in Method *A*. It is essential to use a mechanical stirring device, *F*, to maintain a uniformly distributed temperature. The use of a glass tank or container is required for Method *A*, where it is desired to observe the flow during the test and to record the time for the shellac specimen to flow various stated distances. For Method *B* any suitable container may be used.

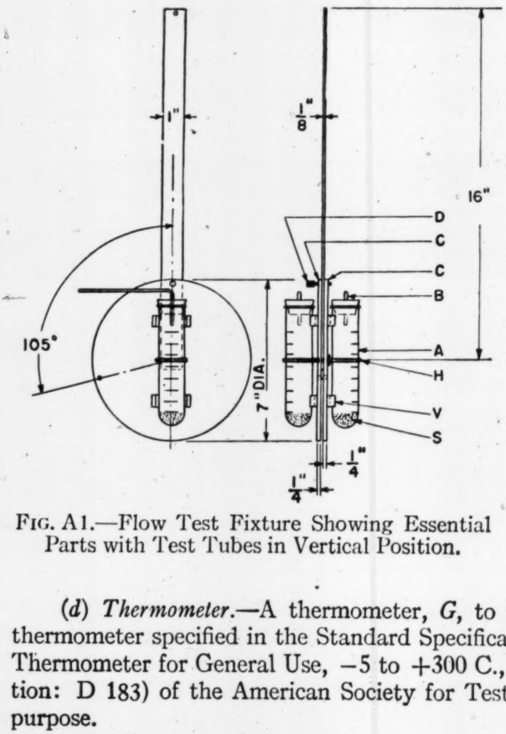

FIG. A1.—Flow Test Fixture Showing Essential Parts with Test Tubes in Vertical Position.

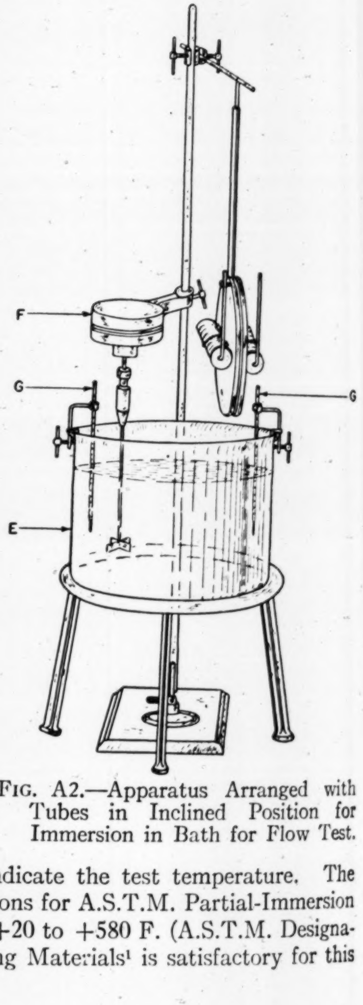

FIG. A2.—Apparatus Arranged with Tubes in Inclined Position for Immersion in Bath for Flow Test.

(*d*) *Thermometer.*—A thermometer, *G*, to indicate the test temperature. The thermometer specified in the Standard Specifications for A.S.T.M. Partial-Immersion Thermometer for General Use, −5 to +300 C., +20 to +580 F. (A.S.T.M. Designation: D 183) of the American Society for Testing Materials[1] is satisfactory for this purpose.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1405.

TENTATIVE METHODS OF TESTING VARNISHED CLOTHS AND VARNISHED CLOTH TAPES USED IN ELECTRICAL INSULATION[1]

A.S.T.M. Designation: D 295 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1928; REVISED, 1929, 1932, 1933, 1934, 1936, 1937.

Scope

1. These methods are intended to apply to varnished cloths and varnished cloth tapes to be used as electrical insulation and are directly applicable to both the "straight-cut" and "bias-cut" materials, unless otherwise stated in the method.

SAMPLING

Selecting Rolls

2. (*a*) Shipments of varnished cloth and varnished cloth tapes shall be sampled as specified in Paragraphs (*b*) and (*c*). The rolls or pads shall be selected in such a manner as to be representative of the shipment.

(*b*) *Cloth.*—One roll shall be selected from each ten rolls in a shipment of full-width varnished cloth. The minimum number of rolls selected from any shipment shall be two.

(*c*) *Tape.*—At least one roll or pad shall be selected from each 1000 rolls or pads in a shipment of varnished cloth tape. The minimum number of rolls or pads selected from any shipment shall be ten.

Selecting Samples

3. (*a*) Except for tape shipped packed in oil, not less than two turns of cloth or six turns of tape from each roll or pad selected for sampling shall be torn off and discarded before the samples are selected. The number of samples required for purposes of test shall then be cut across the entire width of each roll or pad. The length of the samples shall not be less than 91.44 cm. (36 in.) for straight-cut material; from seam to seam for sewed bias-cut material; and from jointed-selvage to jointed-selvage for seamless bias-cut material.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

NOTE 1.—Specimens of bias-cut materials cut from the above samples shall exclude seams and jointed-selvages from the test areas.

NOTE 2.—If it is desired to test the seams or jointed-selvages for tensile breaking strength, additional specimens will have to be cut so that the seams or jointed-selvages are in the center of the specimens.

(*b*) From shipments such as sheets of cloth or strips of tape, the samples shall be representative of the shipment and shall be taken in accordance with Paragraph (*a*).

CONDITIONING

Conditioning

4. (*a*) The roll of cloth or tape from which specimens are to be cut shall be conditioned for a minimum of 2 hr. at a room temperature between the limits of 20 to 30 C., no humidity control.

(*b*) Specimens for tensile breaking strength, elongation, and dielectric strength "as received" shall be removed from the roll as required and tested immediately.

(*c*) If the room humidity is above 65 per cent, the roll of tape shall be kept in a cabinet whose relative humidity is maintained between 60 to 65 per cent.

(*d*) In the event of dispute, the test specimens shall be conditioned for 48 hr. at a temperature between 20 to 30 C. and at a relative humidity between 60 to 65 per cent.

THICKNESS

Apparatus

5. The thickness shall be measured in accordance with Method B of the Tentative Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374 – 36 T) of the American Society for Testing Materials.[1]

Specimens

6. Specimens for test shall consist of samples selected in accordance with Section 3.

Procedure

7. In making thickness measurements, only one layer of material shall be used.

Number of Measurements

8. (*a*) In the case of cloths, ten measurements equally spaced across the width of the specimen shall be taken and the thickness of the cloth shall be the average of the ten measurements.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1026 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1147.

(*b*) In the case of tapes, unless otherwise specified, ten measurements equally spaced along the length of each specimen shall be taken and the thickness of the tape shall be the average of the ten measurements.

NOTE.—The average thickness, as obtained in Section 15 for the dielectric strength test, may be conveniently used instead of the separate test described above.

Report

9. The report shall include the average, maximum, and minimum thicknesses in inches.

TENSILE BREAKING STRENGTH

Apparatus

10. (*a*) A testing machine[1] of the dead-weight pendulum type shall be used. The machine shall preferably be power driven.

(*b*) The capacity of the machine shall not exceed 113 kg. (250 lb.).

(*c*) The machine shall be graduated to read 1 lb. or 1 kg. or less per scale division for testing specimens breaking at 22.7 kg. (50 lb.) or over, and to 0.5 lb. or 0.5 kg. or less for testing specimens breaking under 22.7 kg. (50 lb.).

Test Specimens

11. Specimens cut from the full width cloth or from tapes over 2.54 cm. (1 in.) in width shall be 2.54 cm. (1 in.) in width and not less than 35.54 cm. (14 in.) in length. For tape having a nominal width of 2.54 cm. (1 in.) or under the specimens shall be of the original width and shall be not less than 35.54 cm. (14 in.) in length.

NOTE.—If it is desired to test the seams or jointed-selvages of bias-cut materials for tensile breaking strength, specimens obtained as described in Note 2 to Section 3 (*a*) shall be clamped in the testing machine so that the seams or jointed-selvages are midway between the two jaws. When testing, all readings obtained when the specimen breaks away from the seam or jointed-selvage shall be rejected.

Number of Specimens

12. (*a*) In the case of straight-cut cloths, ten specimens shall be cut in the direction of the warp threads and ten in the direction of the filling threads from samples selected in accordance with Section 3.

(*b*) In the case of bias-cut cloths, ten specimens shall be cut in the direction of the length from samples selected in accordance with Section 3.

(*c*) In the case of tapes, one specimen shall be cut from each roll selected in accordance with Section 2 (*c*).

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[1] This apparatus is the same as that used for the testing of paper and described in Section 16 of the Tentative Methods of Testing Untreated Paper Used in Electrical Insulation (A.S.T.M. Designation: D 202 - 36 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 933 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1047.

Procedure

13. (*a*) The ratio of the clearance distance between jaws to the width of the specimen shall not be less than 5 to 1 nor more than 10 to 1.

(*b*) The rate of travel of the movable jaw shall be constant and preferably 30.5 cm. (12 in.) per minute, but it may be within the limits of 28 cm. (11 in.) and 33 cm. (13 in.) per minute provided it is constant.

(*c*) All readings obtained when the specimen breaks at or in the jaws shall be rejected.

Report

14. (*a*) The tensile breaking strength of a roll shall be the average of the breaking loads of all the specimens from the roll. The report shall include the average, maximum, and minimum breaking loads in kilograms or pounds together with the width of the specimens and the nominal thickness.

(*b*) In the case of straight-cut cloths, the tensile breaking strength of the warp threads and the filling threads shall be reported separately.

DIELECTRIC STRENGTH

Method

15. (*a*) The dielectric strength shall be determined in accordance with the Tentative Methods of Testing Sheet, Tape, and Molded Insulating Materials for Dielectric Strength (A.S.T.M. Designation: D 149 – 36 T) of the American Society for Testing Materials,[1] except that the voltage on the specimen shall in all cases be increased at the uniform rate of 0.5 kv. per second up to breakdown.

(*b*) Dielectric strength tests may be applied to specimens of the material as received and on specimens from the elongation and resistance to oil and heat tests.

Specimens

16. (*a*) In the case of cloths, a specimen at least 10.16 cm. (4 in.) in width shall be cut diagonally from corner to corner from a sample selected in accordance with Section 3.

(*b*) In the case of tapes, the specimens shall be samples selected in accordance with Section 3.

Report

17. The report shall include the following:

(*a*) The method used for making the dielectric strength tests,

(*b*) The average dielectric strength in volts per mil and any other values of dielectric strength as required by the materials specifications, and

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 918 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1002.

(*c*) The temperature at which the tests were made.

ELONGATION

(*In the Case of Bias-Cut Material*)

Scope

18. This method is intended to determine the effect of mechanical tension on bias-cut varnished cloths and tapes. This test is not applicable to tapes of less than 1.27 cm. (0.5 in.) in nominal width.

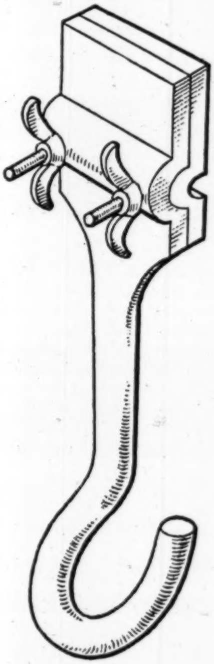

FIG. 1.—Clamp for Elongation Test.

Apparatus

19. The apparatus required for the test shall consist of a pair of clamps for gripping the ends of the specimen. The clamps shall not be less than 5.08 cm. (2.0 in.) in width and one shall be provided with means for attaching to a fixed support and the other with means for affixing suspended weights. See Fig. 1 for suggested form of clamp.

Specimens

20. (*a*) In the case of bias-cut cloths, specimens shall be cut in the direction of the length from the samples selected in accordance

with Section 3. They shall not exceed 3.81 cm. (1.5 in.) in width and shall be at least 91.44 cm. (36 in.) in length.

(*b*) In the case of bias-cut tapes, specimens shall be samples selected in accordance with Section 3 and they shall not exceed 3.81 cm. (1.5 in.) in width. Tapes of greater width than this shall be cut to 3.81 cm. (1.5 in.) or less before testing.

NOTE.—Strips of "Scotch tape" make a satisfactory method for marking the gage length on the elongation test specimens.

Elongation

21. A gage length of 50 cm. shall be marked on the test specimen and the specimen shall be fastened between two suitable clamps so that the gage length is centrally located between the clamps. The clearance distance between each gage line and the adjacent clamp shall be not less than 10 cm.

The test specimen shall be weighted with a specified load, M, (including the weight of the clamp) and at the end of 35 minutes the distance, L, between gage lines shall be measured and the load then removed.

Calculation

22. The percentage elongation is defined by the following formula:

$$\text{Elongation, per cent} = \frac{L - 50}{50} \times 100$$

in which L is in centimeters.

Dielectric Strength

23. After at least 20 min., but not more than 1 hr., following the removal of the load, five puncture voltages shall be taken within the gage length of the specimen in accordance with Section 15, using only Method A of the Tentative Methods D 149 – 36 T.[1] The dielectric strength shall be calculated on the basis of the average thickness of the specimen determined immediately before the dielectric breakdown test.

Report

24. The report shall include the following:

(*a*) the load in pounds per inch width of tape;

(*b*) the percentage elongation;

(*c*) the average dielectric strength in volts per mil after elongation; and

(*d*) the temperature of the room during the elongation test.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 918 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1002.

POWER FACTOR AND DIELECTRIC CONSTANT

Scope

25. This method is intended to determine the power factor and dielectric constant of varnished cloths and tapes at frequencies not exceeding 70 cycles per second.

Definitions and Theory of Test

26. The definitions of dielectric power factor, dielectric constant and related terms, and the theory of test shall be as given in the Tentative Methods of Testing Electrical Insulating Materials for Power Factor and Dielectric Constant (A.S.T.M. Designation: D 150 - 36 T) of the American Society for Testing Materials.[1]

Electrodes

27. Flat, rigid, guarded electrodes, not over 10 sq. in. in area, of such size as to give the bridge sufficient sensitivity to detect readily a change in power factor of 0.0005, shall be used. The specimen shall extend beyond the guard electrode for a distance of at least four times the thickness of the specimen. The pressure shall be not less than 10 nor more than 20 lb. per sq. in.

Test Specimen

28. The test specimen may be tested in one thickness. Petrolatum may be used to avoid ionization. The petrolatum shall have a power factor at 80 C. of not exceeding 0.02 at 60 cycles, or a resistivity not less than 10^{12} ohms per centimeter cube at 80 C.

Conditioning of Specimen

29. The specimens shall be conditioned in one thickness by heating to 105 C. ± 3 C. for 1 hr. without vacuum, and then for 2 hr. at the same temperature in a vacuum having an absolute pressure not exceeding 1 mm. of mercury.

Procedure

30. Tests shall be made at room temperature of 25 to 30 C., and at 80 C. ± 1 C. The conditioned specimens shall be tested within 5 min. after removal from the vacuum container. The voltage gradient shall be 50 v. per mil. The method of measurement shall conform to that described in the Tentative Methods of Testing Electrical Insulating Materials for Power Factor and Dielectric Constant (A.S.T.M. Designation: D 150 - 36 T) of the American Society for Testing Materials.[1]

Number of Tests

31. Three specimens shall be tested and the average shall be taken.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 955 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1069.

Report

32. The report shall include the following:

(*a*) *Test Conditions.*—The frequency in cycles per second, the temperature in degrees Centigrade, relative humidity in per cent, the conditioning of the specimen, size of electrodes, pressure on electrodes, voltage gradient in the dielectric while under test.

(*b*) *Specimen.*—Capacitance of specimen in micro-microfarads, effective area of specimen electrodes, average thickness of specimen between electrodes.

(*c*) Power factor, dielectric constant, and loss factor of each specimen, and the average.

(*d*) Method of measurement.

RESISTANCE TO OIL

Scope

33. This method is intended to determine the effect of oil on the varnish film and dielectric strength of the cloths or tapes.

Specimens

34. One specimen 30.48 cm. (12.0 in.) in length and not exceeding 3.81 cm. (1.5 in.) in width shall be cut from each of the rolls selected in accordance with Section 2.

Procedure

35. The specimens shall be immersed for 15 min. in a specified oil maintained at a temperature of from 97 to 103 C. At the end of this time, the specimens shall be removed from the oil, allowed to cool for at least 30 min. to room temperature and any excess oil removed by placing between blotters without any sliding.

Examination

36. The varnish film shall be examined for disintegration in the oil and flaking either in the oil or on the blotter. Disintegration in the oil may be detected by examination of the used oil for turbidness.

NOTE.—The oil may be considered turbid if a sample of used oil filtered through filter paper is distinctly less transparent than an unfiltered sample of the unused oil when the two samples, in identical containers, are held in front of a diffused light. Flaking along the cut edges of tapes shall not be considered as disintegration of the varnish film.

Dielectric Strength

37. Specimens which have been allowed to cool at least 30 min. may be tested for dielectric strength any time within a period of 4 hr. after removal from the oil. Five puncturing voltages shall be taken on the specimens in accordance with Section 15, using only Method A of the Tentative Methods D 149 – 36 T.[1] The dielectric

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 918 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1002.

strength shall be calculated on the basis of the average thickness of the specimen determined immediately before the dielectric breakdown test.

Report

38. The report shall include the following:

(*a*) The type of oils used (preferably including the flash point determined in accordance with the Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92) of the American Society for Testing Materials).[1]

(*b*) The temperature of the oils.

(*c*) Results of the physical examination of the film (Section 36).

(*d*) The percentage increase in average thickness due to oil tests.

(*e*) The average dielectric strength in volts per mil after the oil tests.

WEIGHT

Weight

39. Specimens of sufficient size to weigh not less than 5 g. (0.18 oz.) shall be accurately weighed on an analytical balance and the weight shall be reported on some commercial unit basis such as, the weight per square yard, the weight per gross yard per 1 in. in width or the weight per roll.

THREADS PER INCH

Count

40. The count or threads per inch of the varnished cloth or varnished cloth tape shall be determined separately on the warp and filling by counting a space of not less than 1 in. (2.54 cm.) in at least five different places in the sample strip (or strips). The average of the five determinations shall be the count.

Report

41. The results of the warp count and the filling count shall be reported separately as threads per inch.

NOTE 1.—The warp threads in straight-cut materials are the threads which are parallel with the length dimension, while in bias-cut materials the warp threads are parallel with the seams and jointed selvages.

NOTE 2.—Before counting black, varnished materials it will be necessary to remove the varnish film with a knife blade or other suitable instrument. Liquid varnish removers are unsuitable for this purpose as they cause a swelling of the fibers and a shrinkage of cloth with a consequent increase in threads per inch; therefore, the films must be removed mechanically.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 892.

TENTATIVE METHODS OF TESTING FLEXIBLE VARNISHED TUBING USED FOR ELECTRICAL INSULATION[1]

A.S.T.M. Designation: D 350 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1932; Revised, 1935, 1937.

Scope

1. These methods cover the testing of flexible varnished tubing (Note) used as electrical insulation for leads on electrical apparatus.

Note.—The term "varnished tubing" as here used refers to braided cotton sleeving coated, or impregnated and coated, with lacquer or a combination of lacquer and varnish or other similar coatings.

SAMPLING

Sampling

2. (*a*) A sufficient number of the pieces of tubing to total not less than 1.83 m. (6 ft.) in length shall be selected from each 305 m. (1000 ft.) in the shipment, but in no case shall less than 1.83 m. (6 ft.) be taken from a shipment and the pieces shall be selected in such a manner as to be representative of the shipment.

(*b*) The number of specimens required for purpose of tests shall be cut from the pieces selected in accordance with Paragraph (*a*) and care shall be taken to select material which is free from abnormal defects such as blisters, wrinkles, cracks, etc.

CONDITIONING

Conditioning

3. (*a*) Test specimens shall be stored for at least 2 hr. in air maintained at a temperature between the limits of 20 and 30 C. (68 and 86 F.), before testing.

(*b*) If the relative humidity of the air is in excess of 55 per cent, then the specimens for the dielectric strength test shall be conditioned in air which is maintained at a relative humidity between the limits of 50 to 55 per cent in a temperature range of 20 to 30 C. (68 to 86 F.) for a period of 48 hr. If a conditioning cabinet is used, one specimen at a time shall be withdrawn and tested immediately for dielectric strength.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

NOTE.—A saturated solution of sodium-dichromate is a suitable humidifying agent for obtaining these limits when used under the conditions specified in Section 11 (*b*) of the Tentative Methods of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation: D 257 - 37 T) of the American Society for Testing Materials.[1]

DIMENSIONS

Gage Rods and Micrometer

4. (*a*) Standard gage rods with hemispherical ends and a 1-in. machinist's type micrometer with ratchet device and having anvil and spindle surfaces 0.25 in. ±0.001 in. in diameter shall be used.

(*b*) The standard gage rods shall be steel and shall have smooth surfaces. A set of two is required for each size of tubing. Each rod in a set shall have a diameter within ±0.0002 in. of the diameters listed as maximum and minimum in the following table:

A.S.T.M. STANDARD SIZES FOR FLEXIBLE VARNISHED TUBING.

	Inside Diameter, In.		
Size	Maximum	Minimum	Nominal
No. 0	0.347	0.325	0.330
No. 1	0.311	0.289	0.294
No. 2	0.278	0.258	0.263
No. 3	0.249	0.229	0.234
No. 4	0.224	0.204	0.208
No. 5	0.198	0.182	0.186
No. 6	0.178	0.162	0.166
No. 7	0.158	0.144	0.148
No. 8	0.141	0.129	0.133
No. 9	0.124	0.114	0.118
No. 10	0.112	0.102	0.106
No. 11	0.101	0.091	0.095
No. 12	0.089	0.081	0.085
No. 13	0.080	0.072	0.076
No. 14	0.070	0.064	0.066
No. 15	0.063	0.057	0.059
No. 16	0.055	0.051	0.053
No. 17	0.049	0.045	0.047
No. 18	0.044	0.040	0.042
No. 19	0.040	0.036	0.038
No. 20	0.036	0.032	0.034

NOTE.—The minimum inside diameters in this table are the same as the diameters of copper wire for corresponding A.w.g. or B. & S. gage sizes.

Test Specimens

5. Test specimens of any convenient length shall be cut from the samples obtained in accordance with Section 2.

Procedure

6. (*a*) *Inside Diameter.*—The minimum gage rod for the size tubing under test shall pass into the specimen for a distance of 5 in. without expanding the wall of the tubing. If the rod has a fairly snug fit, then the specimen has an inside diameter equal to the diameter of the rod. If, however, the minimum gage rod fits loosely, then the maximum gage rod shall

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[1] See p. 1021.

be inserted into the specimen. If the maximum gage rod passes freely into the specimen for a distance of 5 in. with a fairly snug fit or if it expands the wall of the specimen, then the tubing shall be considered to be of that size which falls within the limits of the maximum and minimum inside diameters as represented by the gage rods.

(*b*) *Wall Thickness.*—A standard gage rod which will pass freely into the tubing shall be inserted in the specimen. The micrometer shall then be applied over the specimen, and a reading taken by following the procedure specified in Method A of the Tentative Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374 - 36 T) of the American Society for Testing Materials.[1] The average of five micrometer readings shall be obtained. The wall thickness shall be considered to be one half the difference between the average micrometer reading and the diameter of the gage rod.

(*c*) *Outside Diameter.*—The outside diameter of the specimen shall be considered to be the sum of the estimated inside diameter and twice the wall thickness (Paragraph (*b*)). The inside diameter shall be estimated from the fit of the maximum and the minimum gage rods (Paragraph (*a*)).

Report

7. The report shall include the following:
   (*a*) The size of the tubing,
   (*b*) The wall thickness in inches, and
   (*c*) The outside diameter in inches.

DIELECTRIC STRENGTH

Test Procedure

8. The dielectric strength of the tubing shall be determined in accordance with the short-time dielectric strength test as described in Sections 2 and 9 of the Tentative Methods of Testing Sheet, Tape, and Molded Insulating Materials for Dielectric Strength (A.S.T.M. Designation: D 149 - 36 T) of the American Society for Testing Materials.[2]

Test Specimens and Electrodes

9. (*a*) A straight standard annealed copper wire shall serve as the inner electrode in a test specimen of convenient length. The nearest A.w.g. size of wire shall be selected which will fit snugly without stretching the tubing. The specimen shall be applied over the electrode wire with a straight motion and without twisting the specimen or without causing it to "bunch."

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1026 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1147.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 918 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1002.

The wire shall be free of bends to avoid a binding action on the tubing as it is being slipped onto the wire.

(*b*) After the tubing has been placed on the copper wire, strips of metal foil 2.54 cm. (1 in.) in width shall be tightly wrapped on the outside of the specimen at a distance of not less than 7.62 cm. (3 in.) apart and at least 7.62 cm. (3 in.) from each end. The metal foil shall have a thickness of not to exceed 0.0005 in. and the first turn of the foil applied to the tubing shall be wiped snugly against the tubing. Two more turns of the foil shall then be wound over this first turn, leaving a free end of ½ in. to which the electrical connection shall be made during the test.

NOTE.—The standard gage rods should never be used as the inner electrode. The arc which forms when the tubing punctures will invariably raise a pimple of melted metal on the surface of the rod, spoiling it for use as a standard.

Test Conditions

10. (*a*) The test voltage shall be applied between the copper wire and each metal foil strip in turn.

(*b*) The tests shall be made in air with the specimen at room temperature.

(*c*) The dielectric strength shall be the average of ten puncturing voltages.

Report

11. The report shall include the following:

(*a*) Total volts at each puncture,
(*b*) The average, maximum, and minimum puncturing voltages,
(*c*) The room temperature, and
(*d*) The relative humidity, in per cent.

HEAT ENDURANCE

Test Specimens

12. Not less than ten test specimens, each 15.24 cm. (6 in.) in length shall be cut from the samples obtained in accordance with Section 2.

NOTE.—If the heat endurance is in excess of 120 hr., additional specimens will have to be cut.

Procedure

13. (*a*) The specimens shall be placed in a uniformly heated electric oven of the analytical type in which the temperature is maintained between the limits of 105 and 110 C. (221 and 230 F.). Two specimens shall be removed at the end of 24 hr. and every 24 hr. thereafter until failure occurs. After cooling to room temperature of approximately 20 C. (68 F.), each specimen shall be bent through 180 deg. over a rod having a diameter as shown in the following table:

SIZE	DIAMETER OF TEST ROD, IN.
Nos. 0 to 5, incl	$\frac{5}{16}$
Nos. 6 to 11, incl	$\frac{1}{4}$
No. 12 and smaller	$\frac{1}{8}$

(*b*) The number of hours of baking at which first cracking across the specimen occurs shall be noted and reported. Any side cracking occurring because of the flattening of the specimen on the rod shall be disregarded.

SMOLDERING

(In the case of Grades A and B tubing)

Apparatus

14. The apparatus required for the smoldering test shall be a bunsen burner and a suitable support for holding the specimen.

Test Specimens

15. At least three test specimens 9.16 cm. (4 in.) in length shall be cut from the samples obtained in accordance with Section 2.

Procedure

16. (*a*) A gage length of 2.54 cm. (1 in.) shall be marked on the test specimen at least 2.54 cm. (1 in.) from the end of the specimen. The specimen shall be held mechanically in a horizontal position in still air.

NOTE.—Any metal rod inserted into the specimen for convenience in supporting it must not enter the region spanned by the gage length.

(*b*) The blue flame of the bunsen burner shall be applied to the unsupported end of the specimen until the specimen is thoroughly ignited. The burner shall then be removed. As soon as the leading edge of the flame of the tubing reaches the first gage mark, the flame shall be extinguished with a puff of breath. When all smoldering and smoking has ceased, the distance from the second gage mark to the edge of the charred varnish shall be measured. This measured distance shall be subtracted from 2.54 cm. (1 in.) and the difference shall be considered the distance of smoldering. If the smoldering extends beyond the second gage mark, the smoldering distance shall be reported as over 2.54 cm. (1 in.).

Report

17. The distance of smoldering to the nearest 1.5 mm. ($\frac{1}{16}$ in.) shall be reported.

RATE OF BURNING

(In the case of non-flammable or flame-resistant tubing, Grade C)

Apparatus

18. The apparatus required for the rate of burning test shall be a bunsen burner, a stop watch, and a suitable support for holding the specimen.

Test Specimens

19. At least three test specimens 9.16 cm. (4 in.) in length shall be cut from the samples obtained in accordance with Section 2.

Procedure

20. (*a*) A gage length of 2.54 cm. (1 in.) shall be marked on the test specimen at least 2.54 cm. (1 in.) from the end of the specimen. The specimen shall be held mechanically in a horizontal position in still air.

NOTE.—Any metal rod inserted into the specimen for convenience in supporting it must not enter the region spanned by the gage length.

(*b*) The blue flame of the bunsen burner shall be applied to the unsupported end of the specimen until the specimen ignites. The burner shall then be removed and the time in seconds for the leading edge of the flame to travel over the gage length of 2.54 cm. (1 in.) shall be observed with the stop watch. The rate of burning shall be defined as the number of seconds required to burn a length of 2.54 cm. (1 in.) of the specimen.

Report

21. The rate of burning for each specimen shall be reported.

TENTATIVE SPECIFICATIONS
FOR
RUBBER GLOVES FOR ELECTRICAL WORKERS ON APPARATUS OR CIRCUITS NOT EXCEEDING 3000 VOLTS TO GROUND[1]

A.S.T.M. Designation: D 120 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1923; REVISED AND REISSUED AS TENTATIVE, 1937.[2]

SPECIFICATIONS

Scope

1. These specifications cover electrical workers' rubber gloves of two classes, as follows:

Class A.—Gloves intended for use without external protection.

Class B.—Gloves intended for use with external protection of leather or other material.

Manufacture

2. The gloves shall be made by the "dipping" or other seamless process, of high grade rubber or rubber compound properly vulcanized. Unless otherwise specified, cuff edges shall be finished with a roll or a reinforcing strip of rubber.

Electrical Properties

3. (*a*) *Voltage and Leakage Tests.*—Each glove shall withstand an alternating potential of 10,000 **v.** (r.m.s. value) for 3 min. At 60 cycles the leakage current shall not exceed 10 ma. (total current) at any time during the test. If tested at any other frequency (x cycles) the permissible current leakage (I_x) may be determined by the following formula:

$$I_x = 10 \frac{x \text{ cycles}}{60 \text{ cycles}}$$

(*b*) *Dielectric Strength.*—Each glove shall be capable of withstanding the application of the following voltage (r.m.s. value) for 1 min., at 60 cycles, without showing a leakage current in excess of that given in the following:

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

These specifications are in effect a revision of, and replace the former Standard Specifications for Rubber Gloves for Electrical Workers on Apparatus or Circuits not Exceeding 3000 Volts to Ground (A.S.T.M. Designation: D 120 - 23), which standard was accordingly discontinued in 1937.

[2] Accepted for puplication as tentative by Committee E-10 on Standards, August 26, 1937.

	VOLTS	MILLIAMPERES
Class A	16 000	18
Class B	14 000	14

Physical Properties

4. A test specimen cut from any glove shall conform to the following requirements as to physical properties:

(*a*) *Tensile Strength.*—The tensile strength shall be not less than 1800 lb. per sq. in.

(*b*) *Elongation.*—The total elongation in 1 in. at rupture shall be not less than 600 per cent (1 in. stretched to 7 in.).

(*c*) *Set.*—The set in 1 in. following an elongation of 500 per cent (1 in. stretched to 6 in.) shall not exceed 0.25 in.

Accelerated Aging

5. (*a*) After being subjected to a temperature of 158 F. ± 2 F. (70 C. ± 1 C.) in circulating air for 7 days, the tensile strength and elongation shall be not less than 80 per cent of the original.

(*b*) After being subjected to a temperature of 158 F. ± 2 F. (70 C. ± 1 C.) in oxygen at 300 lb. per sq. in. pressure for 4 days, the tensile strength and elongation shall be not less than 80 per cent of the original.

Size and Length

6. (*a*) Size shall be expressed in inches as measured around the palm at the base of the knuckles. The permissible variation in size shall be $\pm\frac{1}{4}$ in.

(*b*) Length shall be expressed in inches as measured from the tip of the second finger to the edge of the cuff. Unless otherwise specified, the length shall be 14 in. The permissible variation in length shall be $\pm\frac{1}{2}$ in.

Thickness

7. The respective thicknesses of the two classes of gloves in inches shall be within the following limits:

	PART OF GLOVE OTHER THAN CROTCH		CROTCH	
	MAXIMUM	MINIMUM	MAXIMUM	MINIMUM
Class A	0.065	0.050	0.065	0.038
Class B	0.050	0.035	0.050	0.026

Workmanship and Finish

8. Gloves shall be free from patches, blisters, pinholes, cracks, protuberances, embedded foreign matter or other physical defects which can be detected by thorough surface inspection.

Marking

9. Unless otherwise specified each glove shall be marked with the manufacturer's name and the size of glove. Such marking shall be con-

fined to the back of the glove and shall be so done as not to injure the glove. Indelible and non-conducting ink shall be used.

Packing

10. Each pair of gloves shall be packed in an individual stiff paper box of sufficient strength to properly protect the gloves. The end of the box shall be marked with the glove size and in the case of Class B gloves with the words "For use with Protectors."

Guarantee

11. The manufacturer shall replace, without charge to the purchaser, gloves which at any time within a period of eight months from the date of initial test, fail to pass the tests herein prescribed; provided the gloves have been properly stored in their original boxes.

NOTE.—"Proper storage" is to be interpreted to mean that gloves are not to be stored directly above or in proximity to steam radiators or other sources of artificial heat. It is desirable that the ambient temperature shall not exceed 90 F. (32 C.).

Inspection

12. Inspection and test unless otherwise specified shall be made within two weeks of receipt by the purchaser at his own expense at such place as he may designate.

Rejection

13. (*a*) Each glove that fails to meet the requirements of Section 3 (*a*) and (*b*) or Section 8 shall be rejected. Rejection of gloves failing to meet the requirements of Section 6, 9 or 10, is optional with the purchaser.

(*b*) If any of the measurements of thickness made as prescribed in Section 16 or any additional measurements which the purchaser may elect to make fall below the minimum thickness specified in Section 7, the glove shall be rejected. If any of the measurements exceed the maximum thickness specified, rejection is optional with the purchaser.

(*c*) If rejections under Paragraphs (*a*) and (*b*) exceed 25 per cent of the shipment the whole shipment may be rejected without further tests.

(*d*) If 25 per cent or more of the gloves subjected to the test prescribed in Section 3 (*b*) fail to pass the test, the entire shipment shall be rejected.

(*e*) If the average of the results for either the tensile strength, elongation, or set tests made as prescribed in Section 26 fail to meet the requirements of Sections 4 and 5, the entire shipment shall be rejected.

Marking Rejected Gloves

14. All rejected material shall be returned to the manufacturer without being defaced by rubber stamp or other permanent marking, except in gloves which have been punctured on electrical tests. These shall be stamped, punched or cut to indicate that they are unfit for electrical use.

METHODS OF TESTING

Order of Testing

15. The order of procedure in testing rubber gloves shall preferably be as follows:[1]

(*a*) Inspection of inside and outside surfaces (Section 8),
(*b*) Measurement of length (Section 6 (*b*)),

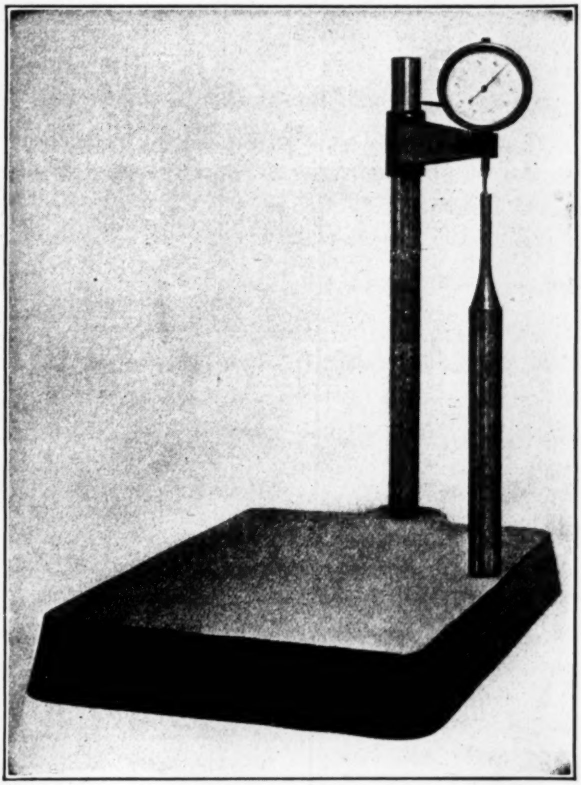

FIG. 1.—Dial Micrometer.

(*c*) Measurement of size (Section 6 (*a*)),
(*d*) Measurement of thickness (Section 7),
(*e*) Electrical test (Section 4 (*a*)),
(*f*) Dielectric strength (Section 4 (*b*)), and
(*g*) Physical properties (Section 5).

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[1] This procedure has been found by experience to minimize the expense of the inspection and testing particularly where an appreciable number of gloves fail to meet the requirements as to workmanship or dimensions.

Thickness Measurement

16. (*a*) *Apparatus.*—The thickness measurements shall be made with any micrometer graduated to mils or tenths of mils having a ratchet friction or spring attachment. The tension on the friction attachment shall be so adjusted that when the spindle has come to a stop, the specimen can be moved between the spindle and anvil without causing any appreciable stretching of the rubber. Dial-type micrometers graduated to mils and mounted in a manner similar to that shown in Figs. 1 and 2 are particularly convenient for making all of these measurements. In both cases the anvil is about $\frac{1}{4}$ in. (6.4 mm.) in diameter and the foot of the spindle about $\frac{1}{8}$ in. (3.2 mm.) in diameter.

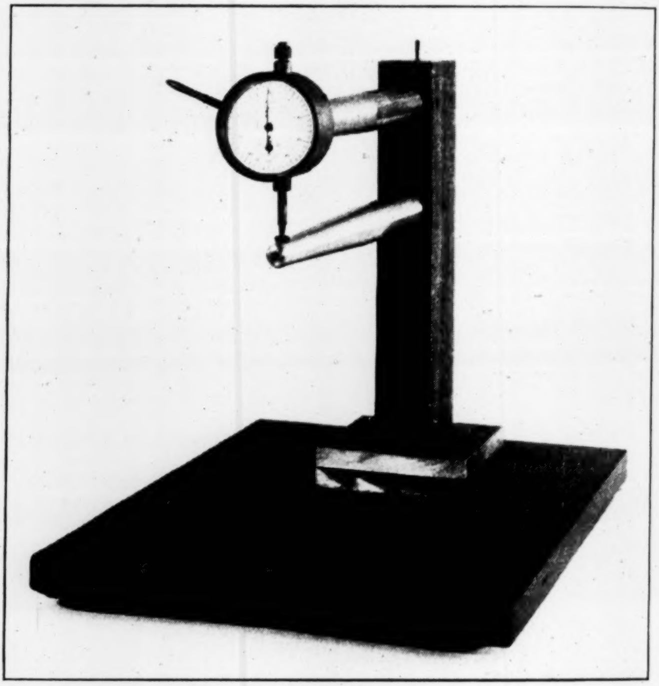

FIG. 2.—Dial Micrometer.

(*b*) *Procedure.*—The thickness of each glove shall be measured at not less than four points on the face and four points on the back. Measurements shall be made at one or more points in the crotch of the thumb and index finger and in the crotches between the fingers.

Electrical Tests

Leakage Test

17. Each glove shall be tested for leakage in fresh water at normal

room temperature (60 to 90 F. (15 to 32 C.)) by immersing it in the water to within about $1\frac{1}{2}$ to 2 in. of the edge of the cuff and filling the glove with water up to the same point. The water inside the glove and that outside the glove form the test electrodes. These are conveniently connected to the testing transformer by means of a chain suspended in the glove and by direct connection to the containing vessel.

Source of Test Voltage

18. The testing voltage shall be obtained from a testing equipment which shall be capable of delivering not less than $\frac{1}{10}$ kva. for each glove tested but in no case shall the rating of the testing transformer be less than 2 kva. The frequency of the testing voltage shall be not more than 65 cycles.

Application of Test Voltage

19. The potential shall be applied at a low value and gradually and steadily raised at a rate of approximately 1000 v. per sec. until the prescribed testing voltage is reached.

Test Period

20. The test period shall be counted from the instant when the prescribed testing voltage is reached.

Voltage Regulation

21. (*a*) The method of regulating the test voltage shall be such that the wave shape of the voltage produced does not have a deviation factor larger than 10 per cent.

> The deviation factor of a wave[1] is the ratio of the maximum difference between corresponding ordinates of the wave and of the equivalent sine wave to the maximum ordinate of the equivalent sine wave when the waves are superposed in such a way as to make this maximum difference as small as possible.

(*b*) Acceptable methods of voltage regulation include the following:

(*1*) Field regulation of the alternator supplying the transformer,
(*2*) Induction-type regulator, and
(*3*) Variable-ratio-transformer type of regulator.

Voltage Measurement

22. The testing voltage shall be measured by one of the following methods:

(*a*) Any properly calibrated commercial type of alternating-current voltmeter connected to the low-tension side of the transformer in conjunction with the ratio of transformation of the transformer provided that the ratio is definitely known for all test conditions.

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[1] Standard No. 7, Alternators, Synchronous Motors and Synchronous Machines in General, Par. 7-650 Deviation Factor of Wave Defined, Am. Inst. Electrical Engrs., December, 1927.

(*b*) A calibrated potential transformer with a voltmeter.

Leakage Current Measurement

23. The leakage current shall be measured directly by inserting a milliammeter in series with each individual glove in turn.

NOTE.—It is customary to make high voltage tests of this character with one end of the circuit grounded, the containing vessel usually being the grounded end of the circuit. The milliammeter should therefore be connected in at the grounded end of the circuit and shunted by a short-circuiting automatic self-closing switch which will therefore always keep the circuit closed except at the instant of reading and thus maintaining an uninterrupted ground.

Dielectric Strength Test

24. (*a*) Not less than ten per cent of the gloves which have passed all of the tests previously prescribed herein shall be selected at random and tested for compliance with Section 3 (*b*).

(*b*) One per cent of the gloves (in no case less than two gloves) which have passed all the tests previously prescribed herein shall be selected at random and tested for ultimate dielectric strength. The test shall be made in accordance with the general method described in Sections 17 to 23 inclusive. The potential shall be raised steadily and gradually at a rate of approximately 1000 v. per sec. until failure occurs, the voltage at failure being recorded.

Physical Tests

Test Specimens

25. Four or more test specimens shall be cut from each glove tested under Section 24 (*b*). The shape of these specimens shall be in substantial accord with that prescribed in the Tentative Methods for Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412 – 36 T) of the American Society for Testing Materials,[1] the essential dimensions of this specimen are as follows:

Total length $4\frac{3}{8}$ in.

Width 1 in. with a reduced section at the center, $\frac{1}{4}$ in. wide and about 1 in. long with a radius of curvature between the ends and the reduced section of $\frac{9}{16}$ in.

Tensile Strength, Elongation and Set Tests

26. The tests for tensile strength, elongation and set shall be made in accordance with the Tentative Methods for Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412 – 36 T) of the American Society for Testing Materials.[1] The essential features of these methods are as follows:

(*a*) Temperature of testing room and of specimens, between 70 and 90 F.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1105 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1266.

(*b*) Rate of separation of jaws of testing machine, 20 in. per min.

(*c*) Tensile strength and elongation shall be made on one specimen and set test on a separate specimen.

(*d*) Set shall be made by stretching the 1-in. gage length to 6 in., holding for 10 min. and measuring the distance between the bench marks 10 min. after release.

Accelerated Aging

27. The tests for accelerated aging of the rubber compound shall be made in accordance with the Tentative Methods of Test for Accelerated Aging of Vulcanized Rubber (A.S.T.M. Designation: D 428 – 36 T) of the American Society for Testing Materials.[1]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1113 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1274.

TENTATIVE SPECIFICATIONS
FOR
INSULATED WIRE AND CABLE: CLASS AO, 30 PER CENT HEVEA RUBBER COMPOUND[1]

A.S.T.M. Designation: D 27 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1916; REVISED, 1921, 1928, 1931, 1933, 1934, 1935, 1936, 1937.[2]

Scope

1. These specifications cover the conductors, insulation, cable tape, cotton braid and lead sheaths of Class AO rubber insulated wire and cable. The design and factor of safety depend upon the service conditions and shall be at the option of the purchaser.

(*A*) CONDUCTOR

MANUFACTURE

Material

2. The conductor shall be of soft annealed copper, properly tinned, and have the properties and characteristics herein required.

Strand

3. Each individual wire of a stranded conductor shall be considered separately and shall be designated as a strand.

Shape

4. Each solid conductor and each strand shall be round and reasonably free from imperfections.

Stranding

5. The stranding shall conform to the requirements of Table I and shall be either standard concentric or flexible as specified in the order.

Density

6. For the purpose of calculating weights, cross-sections, etc., in conductivity determinations, the density of copper shall be taken as 8.89 g. per cu. cm. (See Appendix.)

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

[2] Latest revision accepted by Committee E-10 on Standards, August 26, 1937.

PERMISSIBLE VARIATIONS IN DIMENSIONS

Diameter and Area

7. (*a*) *Permissible Variation.*—When the diameter of solid conductors and strands is specified, the permissible variation from the specified value shall not exceed 1 per cent under, or 3 per cent over for wire 0.010 in. in diameter or larger, and 0.1 mil under or 0.3 mil over for wire less than 0.010 in. in diameter.

When the area of cross-section of stranded conductors is specified, the permissible variation shall not exceed 1 per cent under the specified value.

(*b*) *Method of Gaging Diameter.*—When wire is submitted in coils, each coil shall be gaged in three places, one near each end and one approximately at the middle. When wire is submitted on spools or reels, approximately 12 ft. of the wire shall be reeled off and the wire then gaged in six places between the second foot and the twelfth foot from the end.

TABLE I.—STRANDING OF INSULATED CONDUCTORS.

(*1*) For intermediate sizes use stranding for next larger size.

(*2*) In assembly column, "37 by 19 rope lay," signifies 37 strands of 19 wires assembled like a rope.

(*3*) Flexible stranded Nos. 16 and 18 A. w. g. shall consist of No. 30 A. w. g. wires bunched and bound with a soft cotton wind or other material.

(*4*) Conductors of No. 0000 and smaller are often made solid and this table of stranding should not be interpreted as excluding such practice.

Size of Conductors	Standard Concentric Stranding,[a] Number of Wires	Flexible Stranding	
		Number of Wires	Assembly
2 000 000 cir. mils	127	703	37 by 19 rope lay
1 500 000 cir. mils	91	703	37 by 19 rope lay
1 000 000 cir. mils	61	427	61 by 7 rope lay
500 000 cir. mils	37	427	61 by 7 rope lay
300 000 cir. mils	37	259	37 by 7 rope lay
0 000 A. w. g	19	133	19 by 7 rope lay
1 A. w. g	19	133	19 by 7 rope lay
2 A. w. g	7	133	19 by 7 rope lay
4 A. w. g	7	49	7 by 7 rope lay
8 A. w. g	7	49	7 by 7 rope lay
10 A. w. g	7	37	Concentric
16 A. w. g	7	26	Bunched
18 A. w. g	7	16	Bunched

[a] The standard concentric stranding in this table agrees with Table XII of *Circular No. 31*, "Copper Wire and Cables," U. S. Bureau of Standards; and also Table II of Standard No. 30 "Wires and Cables" of the American Institute of Electrical Engineers.

(*c*) *Calculation of Area.*—The area of cross-section of wire shall be calculated from the average of the measurements of the diameter made in accordance with Paragraph (*b*). The area of cross-section of cable shall be considered to be the sum of the cross-sectional areas of its component wires, when laid out straight and measured perpendicular to their axes.

(*d*) *Rejection.*—A coil or reel shall be rejected if the average diameter obtained from the measurements made in accordance with

Paragraph (*b*), or the area calculated in accordance with Paragraph (*c*), is not within the limits specified in Paragraph (*a*).

A coil or reel shall be rejected if any individual measurement of diameter or of area of cross-section is not within twice the limits of Paragraph (*a*).

PHYSICAL PROPERTIES AND TESTS

Tensile Properties

8. (*a*) Each solid conductor and each strand previous to stranding shall be so drawn and annealed that after tinning it shall conform to the values in Table II.

After stranding, the tensile strength of each strand shall not exceed that specified in Table II by more than 5 per cent and the minimum elongation in 10 in. shall not be less than that specified in Table II by more than 5 per cent.

(*b*) For nominal diameters between listed sizes the requirements shall be those of the next larger sizes indicated in Table II.

Tension Tests

9. (*a*) *Method of Test.*—Tension tests shall be made on fair samples and the elongation shall be determined as the permanent increase in length, due to the breaking of the wire in tension, measured between bench marks originally 10 in. apart. The specimen shall break between the bench marks and not closer than 1 in. to either mark.

TABLE II.—TENSILE REQUIREMENTS OF CONDUCTORS.

Size of Conductor		Maximum Tensile Strength, lb. per sq. in.	Minimum Elongation in 10 in., per cent
Diameter, in.	A.w.g. Number		
0.460 to 0.290	0000 to 1	36 000	30
0.289 to 0.103	2 to 10	37 000	25
0.102 to 0.021	12 to 24	38 500	20
0.020 to 0.012	25 to 28	39 000	15
0.011 to 0.003	29 to 40	40 000	10

(*b*) *Number of Tests.*—Samples shall be taken in accordance with Section 25.

(*c*) *Retests and Rejections.*—If upon testing a sample from a coil, reel or spool of wire, the results are found to be above the stated value for tensile strength or below the stated value for elongation, tests upon two additional samples will be made and the average of the three tests shall determine acceptance or rejection.

Conductivity

10. The conductivity of the conductors after tinning shall be not less than that specified in Table III.

TABLE III.—CONDUCTIVITY REQUIREMENTS.

DIAMETER, IN.	A.W.G. NUMBER	CONDUCTIVITY, PER CENT
0.460 to 0.290	0000 to 1	97.65
0.289 to 0.103	2 to 10	97.15
0.102 to 0.021	12 to 24	96.15
0.020 to 0.012	25 to 28	94.15
0.011 to 0.003	29 to 40	93.15

NOTE.—The above values for conductivity conform to the International Annealed Copper Standard. (See Appendix I.)

Tinning Test

11. (*a*) The continuity of the tin coating of each sample before stranding or insulating shall be determined by the sodium polysulfide test which shall be made in accordance with the requirements of Sections 4 to 10, inclusive, of the Standard Specifications for Tinned Soft or Annealed Copper Wire for Rubber Insulation (A.S.T.M. Designation: B 33) of the American Society for Testing Materials.[1]

(*b*) *Number of Tests.*—Samples shall be taken in accordance with Section 25.

(*c*) *Retests and Rejections.*—If the specimens tested in accordance with Paragraph (*a*) shows any signs of blackening, two more specimens shall be tested. If one of these two additional specimens shows any blackening, that coil or reel shall be rejected. If both specimens are free from any signs of blackening, the coil or reel shall be accepted. If more than 10 per cent of the samples in the entire order fail, all of the wire shall be rejected. If 10 per cent or less of the samples in the entire lot fail, each coil, reel or length may be tested and accepted or rejected upon the results of the individual tests.

(*B*) INSULATION

Workmanship

12. (*a*) Wires and cables ordered under these specifications shall be insulated with Class AO rubber compound. Where performance rubber compound is desired, the order shall be placed under the Tentative Specifications for Insulated Wire and Cable: Performance Rubber Compound (A.S.T.M. Designation: D 353 – 37 T),[2] and where heat-resisting compound is desired, under Tentative Specifications for Insulated Wire and Cable: Heat-Resisting Rubber Compound (A.S.T.M. Designation: D 469 – 37 T)[3] of the American Society for Testing Materials.

(*b*) The insulation shall consist of a properly vulcanized rubber compound which shall be homogeneous in character, tough, elastic and applied concentrically about the conductor and shall fit tightly thereto. Where the insulation is applied in more than one layer, adjacent layers shall be vulcanized into a homogeneous mass.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 669.

[2] See p. 1109.

[3] See p. 1096.

(*c*) *Shielding.*—Braided cables not supported on insulators when operating at 6001 v. and over with the neutral grounded, or 3501 v. and over with the neutral ungrounded shall be shielded or suitably protected against corona. For special installations, such as braided cables connected to an overhead line, shielding may be provided at voltages lower than those given herein.

All lead-covered multiple-conductor cables operating at 6001 v. and over with the neutral ungrounded shall be shielded or suitably protected against corona.

The shielding shall consist of a tinned copper tape at least 3 mils thick or braid of fine tinned copper wires.

(*d*) The insulation of braided wires up to No. 7 A.w.g., inclusive, shall be covered with a single cotton braid. In multiple conductor cables the individual conductors shall not be braid-covered unless so specified. On larger sizes the fibrous covering shall consist of a cable tape and a cotton braid or two cotton braids. The cable tape shall conform to the requirements specified in Sections 52 to 55, inclusive. The cotton braid shall be applied in accordance with Sections 56 to 60, inclusive, and shall be finished in accordance with Sections 61 to 65.

(*e*) Where repairs or joints are made in the insulation, the work shall be done in such manner that the repaired part of the joint, and all parts affected in the process, shall be as strong and durable electrically and mechanically as the remainder of the insulation and shall not exceed the limitations on the thickness specified in Section 17.

MANUFACTURE

Composition

13. (*a*) Class AO rubber compound shall contain exclusively not less than 30 per cent nor more than 33 per cent of the best quality Hevea rubber which has not previously been used in a rubber compound, solid waxy hydrocarbons, suitable mineral matter, antioxidants, organic accelerators, and sufficient sulfur to properly vulcanize the compound.

(*b*) The mineral matter shall be dry.

(*c*) The waxy hydrocarbons used shall be solid at 54° C. and shall be free from saponifiable matter.

(*d*) Contamination of the compound, such as by the use of impregnated tapes, will not excuse the manufacturer from conforming to these specifications.

CHEMICAL PROPERTIES

Quantitative Requirements

14. The insulation after vulcanization shall conform on analysis to the following requirements expressed as percentages by weight of the whole sample:

	MAXIMUM	MINIMUM
Rubber, per cent	33	30
Waxy hydrocarbons, per cent	4	..
Free sulfur, per cent	0.7	..

	30 PER CENT RUBBER[a]	33 PER CENT RUBBER[a]
Saponifiable acetone extract, max., per cent	2.00	2.20
Unsaponifiable resins, max., per cent	1.00	1.10
Chloroform extract, max., per cent	0.90	1.00
Alcoholic potash extract, max., per cent	0.55	0.60
Total sulfur, max., per cent	2.10	2.30

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[a] For percentages between 30 and 33, the limits shall be in proportion to the percentage of rubber found.

Qualitative Requirements

15. (*a*) The acetone solution shall not fluoresce.

(*b*) The hydrocarbons shall be solid, waxy and not darker than a light brown.

(*c*) The chloroform extract (60 ml.) shall not be darker than a straw color.

PHYSICAL PROPERTIES

Physical Properties

16. The insulation shall conform to the following requirements as to physical properties:

Tensile strength, min., lb. per sq. in.		1200
Tensile stress at 200 per cent elongation, min., lb. per sq. in.		300
Elongation at rupture, min., per cent		400
Set in 2-in. gage length, max., in.		¼
Depreciation in tensile strength and elongation after oxygen bomb test, max., per cent		25
Depreciation in tensile strength and elongation after Geer oven test, max., per cent		15
Specific gravity, min.	30 per cent rubber	1.75[a]
	33 per cent rubber	1.67[a]

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[a] For percentages between 30 and 33, the specific gravity limits shall be in proportion to the percentage of rubber found.

THICKNESS

Thickness

17. (*a*) The average thickness of the insulation shall be not less than that given in Table IV. The minimum thickness shall be not

TABLE IV.—THICKNESS OF INSULATION AND ALTERNATING TEST VOLTAGE.

NOTE 1.—Rated circuit voltage of alternating-current circuits is the phase-to-phase voltage.

NOTE 2.—For an intermediate size of wire or an intermediate rated circuit voltage, the thickness and test voltage shall be that for the next larger size or higher rated circuit voltage.

NOTE 3.—For non-leaded submarine cables, $\frac{1}{32}$ in. should be added to the specified respective walls of insulation in this table for all sizes and voltages.

NOTE 4.—Rated circuit voltages from 0 to 5000 v. in this table apply to wire for use on grounded and ungrounded circuits.

NOTE 5.—Above 5000 v. alternating-current, special high-voltage rubber compounds are preferred according to manufacturer's recommendations.

Rated Circuit Voltage	Size of Conductor, A. w. g. Numbers or Circular Mils	Insulation Thickness on each Conductor, sixty-fourths of an inch	Test Voltage
0 to 300	No. 18 to No. 16	2	1 000
0 to 600	No. 14 to No. 9	3	3 000
	No. 8 to No. 2	4	3 500
	No. 1 to No. 0000	5	4 000
	225 000 to 500 000 cir. mils	6	5 000
	525 000 to 1 000 000 cir. mils	7	6 000
	Over 1 000 000 cir. mils	8	7 000
601 to 1000	No. 14 to No. 8	4	5 000
	No. 7 to No. 2	5	6 000
	No. 1 to No. 0000	6	7 500
	225 000 to 500 000 cir. mils	7	9 000
	525 000 to 1 000 000 cir. mils	8	10 000
	Over 1 000 000 cir. mils	9	11 000
1001 to 2000	No. 14 to No. 8	5	6 000
	No. 7 to No. 2	6	7 500
	No. 1 to No. 0000	7	9 000
	225 000 to 500 000 cir. mils	8	10 000
	525 000 to 1 000 000 cir. mils	9	11 000
	Over 1 000 000 cir. mils	9	11 000
2001 to 3000	No. 14 to No. 8	7	9 000
	No. 7 to No. 2	8	10 000
	No. 1 to No. 0000	8	10 000
	225 000 to 500 000 cir. mils	9	11 000
	525 000 to 1 000 000 cir. mils	9	11 000
	Over 1 000 000 cir. mils	10	12 500
3001 to 4000	No. 14 to No. 8	9	11 000
	No. 7 to No. 2	9	11 000
	No. 1 to No. 0000	9	11 000
	225 000 to 500 000 cir. mils	10	12 500
	525 000 to 1 000 000 cir. mils	10	12 500
	Over 1 000 000 cir. mils	11	13 500
4001 to 5000	No. 14 to No. 8	10	12 500
	No. 7 to No. 2	10	12 500
	No. 1 to No. 0000	10	12 500
	225 000 to 500 000 cir. mils	11	13 500
	525 000 to 1 000 000 cir. mils	11	13 500
	Over 1 000 000 cir. mils	12	15 000
5001 to 6000 (grounded)	No. 14 to No. 0000	10	12 500
	225 000 to 1 000 000 cir. mils	11	13 500
	Over 1 000 000 cir. mils	12	15 000
5001 to 6000 (ungrounded)	No. 14 to No. 0000	12	15 000
	225 000 to 1 000 000 cir. mils	12	15 000
	Over 1 000 000 cir. mils	13	16 500
6001 to 7000 (grounded)	No. 8 to No. 0000	11	13 500
	225 000 to 1 000 000 cir. mils	11	13 500
	Over 1 000 000 cir. mils	12	15 000
6001 to 7000 (ungrounded)	No. 8 to No. 0000	14	17 500
	225 000 to 1 000 000 cir. mils	14	17 500
	Over 1 000 000 cir. mils	15	19 000
7001 to 8000 (grounded)	No. 8 to No. 0000	12	15 000
	225 000 to 1 000 000 cir. mils	12	15 000
	Over 1 000 000 cir. mils	13	16 500
7001 to 8000 (ungrounded)	No. 8 to No. 0000	16	20 000
	225 000 to 1 000 000 cir. mils	16	20 000
	Over 1 000 000 cir. mils	17	21 000

less than 95 per cent of the thickness required by Table IV for insulations less than $\frac{4}{64}$ in. in thickness and not less than 90 per cent of the specified thickness for insulations of $\frac{4}{64}$ in. and greater.

(*b*) The thicknesses in Table IV apply to single conductor cable and the individual conductors of multiple conductor cables, leaded or braided, except special applications such as aerial, non-leaded submarine, and vertical riser cable.

TABLE V.—INSULATION RESISTANCE, MEGOHMS-1000 FT. AT 60° F. (15.5° C.).

NOTE 1.—This table is based on a constant, $K = 21\,120$ in the following formula:

$R = K \log_{10} \frac{D}{d}$; where R = resistance in megohms-1000 ft., K = constant.

D = diameter over insulation, on each conductor, and d = diameter over conductor.

NOTE 2.—For an intermediate size, the insulation resistance shall be that of the next larger size.

Size of Conductor, A. w. g. Numbers or Circular Mils	Solid or Stranded	Diameter in Inches	Insulation Thickness in Sixty-fourths of an Inch									
			2	3	4	5	6	7	8	9	10	12
No. 18	Solid	0.040	8600	11 100	....	....	....	....	....	....	....	....
No. 16	Solid	0.051	7300	9 500	....	....	....	....	....	....	....	....
No. 14	Solid	0.064	....	8 300	9900	11 300	12 550	13 650	14 600	15 450	16 250	17 650
No. 12	Solid	0.081	....	7 050	8550	9 850	11 000	12 000	12 900	13 750	14 500	15 850
No. 10	Solid	0.102	....	6 000	7350	8 500	9 600	10 500	11 350	12 100	12 850	14 150
No. 8	Solid	0.128	....	....	6200	7 300	8 300	9 150	9 950	10 650	11 350	12 550
No. 6	Stranded	0.184	....	....	4750	5 600	6 400	7 200	7 900	8 500	9 100	10 200
No. 4	Stranded	0.232	....	....	3900	4 700	5 450	6 100	6 700	7 250	7 800	8 850
No. 2	Stranded	0.292	....	....	3250	3 900	4 550	5 150	5 650	6 150	6 650	7 600
No. 1	Stranded	0.332	....	....	....	3 550	4 100	4 650	5 150	5 600	6 100	6 950
No. 0	Stranded	0.373	....	....	....	3 200	3 750	4 200	4 700	5 150	5 550	6 350
No. 00	Stranded	0.418	....	....	....	2 900	3 400	3 850	4 300	4 700	5 100	5 850
No. 000	Stranded	0.470	....	....	....	2 600	3 100	3 500	3 900	4 250	4 650	5 400
No. 0000	Stranded	0.528	....	....	....	2 400	2 800	3 150	3 550	3 900	4 200	4 900
250 000 cir. mils	Stranded	0.575	....	....	....	....	2 600	2 950	3 300	3 650	4 000	4 600
350 000 cir. mils	Stranded	0.681	....	....	....	....	2 250	2 550	2 850	3 150	3 450	4 000
500 000 cir. mils	Stranded	0.814	....	....	....	....	1 900	2 200	2 450	2 700	2 950	3 450
750 000 cir. mils	Stranded	0.998	....	....	....	....	....	1 800	2 050	2 250	2 500	2 900
1 000 000 cir. mils	Stranded	1.152	....	....	....	....	....	1 600	1 800	2 000	2 200	2 600
1 250 000 cir. mils	Stranded	1.289	....	....	....	....	....	....	1 600	1 800	2 000	2 350
1 500 000 cir. mils	Stranded	1.412	....	....	....	....	....	....	1 500	1 650	1 850	2 150
1 750 000 cir. mils	Stranded	1.526	....	....	....	....	....	....	1 400	1 550	1 700	2 000
2 000 000 cir. mils	Stranded	1.631	....	....	....	....	....	....	1 300	1 500	1 600	1 900

Size of Conductor, A. w. g. Numbers or Circular Mils	Solid or Stranded	Diameter in Inches	Insulation Thickness in Sixty-fourths of an Inch				
			13	14	15	16	17
No. 8	Solid	0.128	13 100	13 600	14 100	14 600	14 900
No. 6	Stranded	0.184	10 650	11 150	11 500	12 000	12 450
No. 4	Stranded	0.232	9 300	9 700	10 100	10 500	10 900
No. 2	Stranded	0.292	7 950	8 400	8 800	9 150	9 500
No. 1	Stranded	0.332	7 350	7 650	8 000	8 400	8 750
No. 0	Stranded	0.373	6 750	7 050	7 400	7 800	8 100
No. 00	Stranded	0.418	6 200	6 500	6 900	7 150	7 500
No. 000	Stranded	0.470	5 700	6 150	6 350	6 650	6 900
No. 0000	Stranded	0.528	5 200	5 500	5 800	6 050	6 350
250 000 cir. mils	Stranded	0.575	4 900	5 200	5 450	5 750	5 950
350 000 cir. mils	Stranded	0.681	4 250	4 500	4 700	4 950	5 200
500 000 cir. mils	Stranded	0.814	3 700	3 950	4 150	4 350	4 550
750 000 cir. mils	Stranded	0.998	3 100	3 300	3 500	3 700	3 900
1 000 000 cir. mils	Stranded	1.152	2 750	2 950	3 100	3 300	3 450
1 250 000 cir. mils	Stranded	1.289	2 500	2 650	2 850	3 000	3 150
1 500 000 cir. mils	Stranded	1.412	2 300	2 450	2 600	2 700	2 950
1 750 000 cir. mils	Stranded	1.526	2 150	2 300	2 450	2 600	2 750
2 000 000 cir. mils	Stranded	1.631	2 050	2 200	2 300	2 450	2 600

NOTE.—In Appendix II will be found an interpretation of the insulation thicknesses given in Table IV.

ELECTRICAL PROPERTIES

High Voltage Test

18. Each coil, reel, or length of wire or cable, after vulcanization shall successfully withstand the application of an alternating voltage of not less than that prescribed in Table IV for a period of 5 minutes. (See Sections 47 and 48.)

Insulation Resistance

19. Each coil, reel, or length of wire or cable after being subjected to the high voltage test specified in Section 18 shall have an insulation resistance not less than that given in Table V.

Moisture Absorption

20. (*a*) The increase in the specific inductive capacity of the insulation shall be used as an indication of the amount of moisture absorbed.

(*b*) The specific inductive capacity after 24-hr. immersion in water shall not exceed six. The increase between the ends of the first and the fourteenth days shall not exceed 20 per cent and that between the ends of the seventh and fourteenth days, 5 per cent.

(*c*) The specific inductive capacity shall be determined at either 1000 or 60 cycles. Where measured at 1000 cycles, the voltage impressed upon the conductor shall not exceed 10 volts. Where measured at 60 cycles, the voltage impressed upon the conductor shall be equivalent to a stress of between 30 to 40 volts per mil of insulation.

(*d*) The measurements shall be made as described in Section 51.

METHODS OF TESTING

CHEMICAL ANALYSIS

Sampling

21. Samples of the vulcanized rubber compound for chemical analysis shall be taken after the successful completion of the physical and electrical tests, and before applying impregnating compounds which may contaminate the rubber. The samples shall be taken at the option of the purchaser. The purchaser shall assure himself that all samples are free from contamination and change due to torch heating. Wherever practicable, samples shall be taken at a distance of at least 3 ft. from the end of the wire.

Procedure

22. The insulation shall be analyzed in accordance with the Complete Procedure as described in the Standard Methods of Chemical Analysis of Rubber Products (A.S.T.M. Designation: D 297) of the American Society for Testing Materials.[1]

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1249.

Rejection and Retests

23. The purchaser may make a chemical analysis on any one of the samples selected as described in Section 21 to determine if the compound conforms to the requirements of Sections 14 and 15. Failure of any sample selected to conform to the requirements shall be sufficient cause for rejecting the wire which that sample represents, except in the case of failure to conform to the requirements for either or both free sulfur and chloroform extract. In case of such failure, the coil from which the sample was taken shall be rejected and two additional samples shall be selected from the remainder of the order. The remainder of the order shall be accepted if both samples so selected conform to the requirements for free sulfur or chloroform extract. The entire order shall be rejected if either sample fails.

SPECIFIC GRAVITY

Specific Gravity

24. The specific gravity shall be determined in accordance with Section 14 of the Standard Methods of Chemical Analysis of Rubber Products (A.S.T.M. Designation: D 297) of the American Society for Testing Materials.[1]

TENSILE STRENGTH, SET AND ELONGATION AT RUPTURE

Sampling for Physical Tests

25. (*a*) Wire up to and including 250,000 cir. mils in size shall be sampled in accordance with the following requirements:

QUANTITY OF WIRE	NUMBER OF SAMPLES
2000 ft. and under	none
2001 to 10,000 ft.	1 for each 2000 ft. or fraction thereof
10,001 to 100,000 ft.	5 plus 1 for each 10,000 ft. additional over 10,000 ft.
100,001 to 500,000 ft.	14 plus 1 for each 25,000 ft. over 100,000 ft.

(*b*) For wire larger than 250,000 cir. mils, one sample shall be selected from quantities between 1000 and 2000 ft. and for larger quantities double those specified in Paragraph (*a*).

Test Specimen

26. (*a*) *Size.*—The test specimen may be the entire section of the insulation in the case of small wires, but in the case of wire and cable of No. 6 A. w. g. and larger or with an insulation thickness greater than $\frac{3}{32}$ in., samples approximately square in section shall be cut from the insulation with a cross-section not greater than 0.025 sq. in. The test specimen shall be as free as possible from surface incisions and imperfections.

NOTE.—The removal of the rubber insulation can be greatly accelerated and in most cases a test specimen which is an entire section can be obtained free from surface incisions and imperfections by means of metallic mercury. The mercury should be introduced at one end of the sample between the insulation and the tinned surface of the conductor, and the sample inclined on a support with the end to which the mercury is applied at the top. The separation of the rubber insulation results from the amalgamation of the tin of the conductor with the mercury. The amalgamation is assisted by first immersing and rubbing the tinning on the exposed end of the conductor in the mercury.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1249.

(*b*) *Condition and Age.*—Specimens shall not be heated, immersed in water or subjected to any mechanical or chemical treatment not specifically prescribed in these specifications. Specimens for accelerated aging tests shall be taken from the completed wire after the application of the specified covering and shall be aged with all such covering removed. No tests shall be made within 24 hours nor later than 60 days after vulcanization unless agreed to by the manufacturer.

(*c*) *Buffing.*—In the event of any irregularities on the surface of the test specimen including corrugations from large strands, it shall be made smooth and of uniform thickness by buffing.

Calculation of Area

27. Calculation of the area of the test specimens shall be made as follows:

(*a*) Where the total cross-section of the insulation is used, the area shall be taken as the difference between the area of the circle whose diameter is the minimum average outside diameter of the insulation and the area of the conductor. The area of a stranded conductor shall be calculated from its maximum diameter.

(*b*) Where a slice cut from the insulation by a knife held tangent to the wire is used, and the slice so cut has the cross-section of a segment of a circle, the area shall be calculated as that of the segment of a circle whose diameter is that of the insulation. The height of the segment is the wall of insulation on the side from which the slice is taken. (The values may be obtained from a table giving the areas of segments of a unit circle for the ratio of the height of the segment to the diameter of the circle.)

(*c*) Where the cross-section of the slice is not a segment of a circle, the area shall be calculated from a direct measurement of the volume or from the specific gravity and the weight of a known length of the specimen having a uniform cross-section.

(*d*) Where a portion of a sector of a circle has to be taken where the conductor is large and the insulation thin, the area shall be calculated as the thickness times the width. (This applies either to a straight test piece or one stamped out with a die, and assumes that corrugations have been removed by buffing.)

(*e*) Where a portion of a sector of a circle has to be taken where the conductor is large and the insulation thick, the area shall be calculated as the proportional part of the area of the total cross-section.

Temperature at Test

28. Physical tests shall be made at a room temperature not less than 65° F. (18° C.) nor more than 90° F. (32° C.) and the test specimen shall have been kept at the room temperature not less than 30 minutes prior to the test.

Tension Testing Machine

29. The tension testing machine shall be power-driven and preferably of the pendulum type. The machine shall be accurate within 1 per cent of the breaking load. A spring balance type of apparatus may be used if provided with a device which will indicate the actual maximum load at which rupture takes place and if provision is made to prevent recoil of the spring.

Tensile Strength Test Procedure

30. The tensile strength test shall be made on a specimen which has not been previously stretched. The specimen shall have a length of not less than 6 in., and shall be marked with gage marks 2 in. apart and placed in the jaws of the testing machine with a maximum distance between jaws of 4 in. The specimen shall be stretched at the rate of 20 in. per minute (jaw speed) until it breaks. The test specimen shall break between the gage marks. The tensile strength shall be calculated upon the area of the original specimen.

Tensile Stress Test

31. The tensile stress test shall be made in conjunction with the tensile strength test by recording the load when the 2-in. gage length marks are 6 in. apart, that is, at 200 per cent elongation. The tensile stress shall be calculated upon the area of the original specimen.

Elongation at Rupture Test Procedure

32. The percentage of elongation at rupture shall be determined simultaneously with the tensile strength and on the same specimen.

The elongation at rupture shall be taken as the distance between gage marks at rupture less 2 in. (the original gage length of the set test specimen). The percentage of elongation at rupture is the elongation in inches divided by the original gage length (2 in.) and multiplied by 100 to express as a percentage.

Set Test Procedure

33. The set test shall be made on a separate test specimen having a length of not less than 6 in. and marked with gage marks 2 in. apart. The specimen shall be placed in the jaws of the testing machine with

a maximum distance between jaws of 4 in. and shall be stretched at the rate of 20 in. per minute (jaw speed) until the gage marks are 6 in. apart. The test specimen shall then be released within 5 seconds and the distance between gage marks shall be determined 1 minute after the beginning of release. The set is the difference between this length and the original 2-in. gage length.

Retests, Physical Properties

34. Where tested in accordance with Sections 30, 31, 32 and 33, if a specimen fails to conform to any one of the requirements for tensile strength, tensile stress, set, or elongation at rupture specified in Section 16, two additional specimens shall be taken from the same sample and tested, and the average of the results shall determine whether the material conforms to the requirements.

AGING TESTS

Sampling for Aging Tests

35. For aging tests the wire shall be sampled as follows:

No samples shall be selected where the quantity is less than 2000 ft. of wire or cable.

For sizes less than 250,000 cir. mils, one sample shall be selected for each quantity ordered between 2000 ft. and 50,000 ft. of wire or cable and one additional sample for each additional 50,000 ft. thereafter.

For sizes 250,000 cir. mils and over, one sample shall be selected for each quantity ordered between 2000 ft. and 25,000 ft. of wire or cable and one additional sample for each additional 25,000 ft. thereafter.

Test Specimens

36. Nine test specimens of similar size and shape shall be prepared from each sample selected in accordance with Section 35, three for the determination of the initial or unaged properties, three for the oxygen bomb test and three for the Geer oven test. One specimen of each three shall be tested and the other two held as spares except that where only one sample is selected all three specimens shall be tested and the average of the results reported.

Aging Test Conditions

37. (*a*) In the case of wire and cable smaller than No. 6 A.w.g. with an insulation thickness less than $\frac{3}{32}$ in., the insulation shall be heated in the oxygen bomb and in the Geer oven without removing the conductor.

(*b*) In the case of wire and cable of No. 6 A.w.g. and larger or with an insulation thickness greater than $\frac{3}{32}$ in., samples approximately square in section shall be cut from the insulation with a cross-section not greater than 0.025 sq. in.

(*c*) Die-cut samples shall be buffed before being subjected to the accelerated aging test wherever the thickness of the sample is $\frac{3}{32}$ in. or greater after buffing.

(*d*) The test specimens shall have no protective covering and shall be suspended vertically in such a manner that they will not come in contact with each other or with the sides of the bomb or oven.

(*e*) Physical tests on both aged and unaged specimens shall be made at the same time. The aged specimens shall have a rest period of not less than 16 hr. nor more than 48 hr. between the completion of the aging and the determination of the physical properties.

Oxygen Bomb Test

38. The test specimens shall be heated in an atmosphere of oxygen at a pressure of 300 lb. per sq. in. at a temperature of from 157 to 159° F. for a period of 96 hours. The weight of the rubber in the bomb shall not be over 2 g. per cu. in. of bomb space. The bomb pressure shall be reduced at a uniform rate, requiring at least two minutes, when specimens are removed. The bomb temperature shall be recorded automatically on a chart.

Gear Oven Test

39. The test specimens shall be heated at a temperature of from 157 to 159° F. for a period of 96 hours in an oven having a free circulation of fresh air. The oven temperature shall be recorded automatically on a chart.

Retests, Aging

40. Should the first set of specimens selected under Section 35 fail to meet the specifications, two other sets of specimens shall be tested and if either of these sets fails, the coils, reels or lengths will be rejected.

Rejection

41. When ten or more samples are selected in any inspection lot, all coils, reels or lengths may be rejected if more than 10 per cent of the samples fail. If 10 per cent or less fail, each coil, reel or length may be tested and shall be accepted or rejected upon the results of such individual tests. Where the number of samples selected in any inspection lot is less than ten, all coils, reels or lengths shall be rejected if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel or length may be tested and shall be accepted or rejected upon the result of such individual tests.

THICKNESS

Apparatus

42. The thickness measurements may be made with any type of micrometer reading to 0.001 in., suitable for measurements of this character.

Number of Measurements

43. Where the lot of wire to be inspected consists of two coils or reels or less, at least one determination of the thickness shall be made on each coil or reel. Where the lot consists of more than two coils or reels and less than 20 coils or reels, at least one determination of the thickness shall be made on each of two coils or reels taken at random. If the lot consists of 20 or more coils or reels, not less than 10 per cent of the coils or reels shall be selected at random and at least one determination of the thickness made on each coil or reel so selected.

TABLE VI.—TEMPERATURE COEFFICIENTS.

Temperature		Temperature Coefficient	Temperature		Temperature Coefficient
Deg. Fahrenheit	Deg. Centigrade		Deg. Fahrenheit	Deg. Centigrade	
46	7.8	0.69	61	16.1	1.03
47	8.3	0.71	62	16.7	1.05
48	8.9	0.73	63	17.2	1.08
49	9.4	0.75	64	17.8	1.11
50	10.0	0.77	65	18.3	1.14
51	10.6	0.79	66	18.9	1.17
52	11.1	0.81	67	19.4	1.20
53	11.7	0.83	68	20.0	1.23
54	12.2	0.85	69	20.6	1.26
55	12.8	0.88	70	21.1	1.30
56	13.3	0.90	71	21.7	1.33
57	13.9	0.92	72	22.2	1.37
58	14.4	0.94	73	22.8	1.40
59	15.0	0.97	74	23.3	1.44
60	15.6	1.00	75	23.9	1.48

Procedure

44. The average thickness of the insulation shall be taken as one-half the difference between the mean of the maximum and minimum diameters measured at any one point and the average diameter of the conductor measured at the same point. The minimum thickness shall be taken as the difference between a measurement made over the conductor plus the thinnest wall and the diameter of the conductor. (The first measurement is made by "slicing off" the heavier side of the insulation.)

In the case of multiple-conductor cable, the measurements shall be made on the individual wires before being cabled.

Rejection

45. If the thickness of the insulation of any coil or reel is found to be less than the specified value, that coil or reel shall be rejected and a thickness measurement on each of the remaining coils or reels shall be made.

ELECTRICAL TESTS

Place of Testing

46. Electrical tests of wire and cable shall be made at the place of manufacture.

High Voltage Test

47. The high voltage test shall be made with alternating potential from a transformer and generator of ample capacity and in no case less than 5 kva. The frequency of the test voltage shall be not greater than 100 cycles and shall have a wave shape approximating as closely as possible a sine curve.

Rate of Application

48. The initially-applied voltage shall be not greater than the rated voltage and the rate of increase shall be approximately uniform and not over 100 per cent in 10 seconds nor less than 100 per cent in 60 seconds.

High Voltage Testing

49. (*a*) *General.*—Where insulated conductors are subjected to a high voltage test in water they shall be immersed in a grounded water bath and tested while still immersed.

(*b*) *Single Conductor.*—Single conductors shall be tested after 12-hr. immersion and without covering except a tape or braid applied before vulcanization.

Rubber-sheathed conductors shall be immersed and tested after the sheath is applied.

Where a metallic sheath is applied, the conductors shall be retested against the sheath.

Single-conductor cables shall be tested between conductor and sheath or water in which they are immersed at the test voltage required by Table IV.

(*c*) *Multiple Conductor.*—The individual conductors of multiple-conductor cables shall be tested before assembling as prescribed in Paragraph (*b*).

After assembly each conductor shall be tested against the adjacent conductors. Where a metallic sheath is applied each conductor shall in addition be tested against the sheath.

Cables having a rubber or other non-metallic waterproof sheath shall be immersed for 12 hr. and the conductors tested against ground. Cables covered with cable tape and braid or double braid with a weather-resistant finish shall not be immersed for testing.

Multiple-conductor cables after assembly shall be tested at the test voltage required by Table IV.

Insulation Resistance

50. (*a*) The insulation resistance shall be measured after the high-voltage test has been made and while the wire or cable is still immersed in water. The measurement shall be made after one-minute electrification with a continuous e.m.f. of from 100 to 500 volts, the conductor being maintained negative to the water. The temperature of the water shall be within the limits given in Table VI.

Temperature Coefficient

(*b*) If the temperature at which the insulation resistance was measured differs from 60° F. (15.5° C.), the resistance shall be reduced to that at 60° F. (15.5° C.) by multiplying the measured value by the coefficient in Table VI corresponding to the temperature at which the measurement was made.

Moisture Absorption Test

51. (*a*) A 15-ft. sample of the insulated wire or cable shall be taken after vulcanization and prior to the application of any coverings except tape applied before vulcanizing. Such cable tape shall be removed before making the moisture absorption test. The middle 10 ft. of the sample shall be immersed in distilled water for a period of 14 days with the $2\frac{1}{2}$-ft. portion at each end kept above the water as leakage insulation. The three measurements specified in Section 20 shall be taken with the water at the same temperature. The water shall be maintained at room temperature, but not less than 70 F. (21 C.).

(*b*) The specific inductive capacity of the insulation shall be determined at commercial frequencies and shall be calculated as follows:

$$\text{Specific inductive capacity} = 13{,}600\ C \log_{10} \frac{D}{d}$$

where C = the capacity in microfarads of 10 ft. of sample;
D = the diameter over insulation; and
d = the diameter over conductor.

The moisture absorption test shall be made only when requested by the purchaser.

(*C*) CABLE TAPE

Material

52. Cable tape shall be made from cotton cloth having a weight of not less than 1 lb. per 4 yd. based on a width of 36 in. and not less than 56 by 60 picks per inch, and shall be frictioned on both sides and thoroughly filled with a rubber compound. The nominal thickness shall be 0.012 in. and not less than 0.011 in.

TABLE VII.—WIDTH AND OVERLAP OF RUBBER-FILLED CABLE TAPE.

Diameter Over Insulation, in.	Maximum Width of Tape, in.	Minimum Overlap, in.
2.00	5	½
1.75	4½	½
1.50	4	½
1.25	3½	½
1.15	3¼	½
1.00	3	½
0.88	2¾	⅜
0.75	2⅝	⅜
0.62	2	⅜
0.50	1¾	⅜
0.38	1½	¼
0.31	1¼	¼
0.25	1	¼
0.19	⅞	3/16
0.16	¾	3/16

TABLE VIII.—TANGENT OF ANGLE OF BRAID.

Diameter Under Braid, in.	Value of Tangent	Corresponding Braid Angle, deg.
0 to 1.00	0.700	35
1.001 to 1.500	0.839	40
1.501 and over	1.000	45

Application

53. The tape shall be applied helically and may be applied before vulcanization. The maximum width and minimum overlap shall conform to the values in Table VII.

Intermediate Diameters

54. For intermediate diameters the requirements shall be those of the next smaller diameter indicated in Table VII.

Number of Tests

55. Samples shall be taken at the option of the purchaser.

(*D*) COTTON BRAID

Braids

56. (*a*) Two types of cotton braid are covered in these specifications, namely, standard braid and heavy braid, as follows:

Standard Braid is designed for indoor service and installation in conduits.

Heavy Braid is designed for outdoor and rough service.

(*b*) Unless otherwise specified, standard braid shall be supplied under these specifications.

Material

57. Cotton braid shall be closely woven from cotton thread having not less than two plies.

TABLE IX.—MINIMUM THICKNESS OF STANDARD BRAID.

DIAMETER UNDER BRAID, IN.	MINIMUM THICKNESS OF BRAID, IN.	CORRESPONDING MINIMUM SIZE AND PLY OF COTTON YARN
0 to 0.200	0.016	30/2
0.201 to 0.350	0.017	26/2
0.351 to 0.800	0.020	20/2
0.801 to 1.500	0.026	12/2
1.501 to 3.000	0.032	8/2

TABLE X.—MINIMUM THICKNESS OF HEAVY BRAID.

DIAMETER UNDER BRAID, IN.	MINIMUM THICKNESS OF BRAID, IN.	CORRESPONDING MINIMUM SIZE AND PLY OF COTTON YARN
0 to 0.200	0.020	20/2
0.201 to 0.300	0.022	16/2
0.301 to 0.600	0.026	12/2
0.601 to 1.000	0.032	8/2
1.001 to 1.500	0.037	6/2
1.501 to 2.000	0.045	4/2
2.001 to 3.000	0.056	4/3

Construction

58. (*a*) The braid shall be so constructed that the tangent of the angle between the cotton thread and the side of the wire shall be not less than that specified in Table VIII.

(*b*) The thickness of standard braid shall be not less than that specified in Table IX and that of heavy braid not less than that specified in Table X.

(*c*) The number of carriers and the number of ends per carrier used in applying the braid shall be optional providing all other limitations of Sections 57 and 58 are met. However, the picks per inch shall be not less than the value as calculated by the following formula:

$$P = \sqrt{\left[\frac{1}{tE + M}\right]^2 - \left[\frac{C}{2\pi\,(D + T)}\right]^2}$$

where P = the picks per inch;

T = the thickness of braid, in inches; $T = 2t$

t = the thickness of one end of cotton, in inches; $t = \dfrac{0.0279\ K}{\sqrt{S}}$

K = cabling factor for plied yarn; values of K are: $\begin{cases} \text{2 ply} = 1.60 \\ \text{3 ply} = 2.00 \\ \text{4 ply} = 2.36 \end{cases}$

S = the cotton ply, size or number;

E = the number of ends per carrier;

C = the number of carriers;

D = the diameter under braid, in inches;

M = the space between each set of carrier threads, in inches; $M = X\,(0.011 + 7.0\ t^2E^2)$

X = the closeness constant: Values of X:

	Diameter Under Braid, in.
1.1......	0 to 0.500
1.0......	0.501 and over

For convenience in calculating the more usual braid constructions, the formula for picks may be reduced to:

$$P = \sqrt{Y - \frac{Z}{(D + T)^2}} \quad \text{where } Y = \frac{1}{(tE + M)^2} \quad \text{and } Z = \left(\frac{C}{2\pi}\right)^2$$

Values of Y for some of the most commonly used cottons are:

Size of Yarn	Thickness of Braid, *T*	Values of *Y*										
		For Diameters Under 0.500 in.				For Diameters 0.501 in. and Over						
		2 End	3 End	4 End	5 End	2 End	3 End	4 End	5 End	6 End	7 End	8 End
30/2	0.0163................	1081	592	358	233	....	....	....	....	....	....	....
26/2	0.0175................	982	526	312	220	....	....	....	....	....	....	....
24/2	0.0182................	930	492	290	185	....	....	....	....	....	....	....
20/2	0.0200................	807	416	241	151	878	446	259	163	....	....	....
16/2	0.0223................	683	339	193	118	738	365	207	127	....	....	....
14/2	0.0238................	616	302	168	103	664	324	180	111	....	....	....
12/2	0.0258................	538	257	142	85	582	276	152	92	59	40	28
10/2	0.0282................	....	....	....	....	497	231	125	74	48	32	22
8/2	0.0316................	....	....	....	....	407	183	96	56	35	23	16

Values of Z for the standard braiders are:

NUMBER OF CARRIERS	VALUES OF *Z*
12..	3.65
16..	6.49
20..	10.13
24..	14.59
36..	32.80
48..	58.40

(*d*) For braid over twin wires use 1.64 times the conductor diameter to determine the proper braid.

(*e*) Where two braids are specified, the outer braid shall be determined by the diameter over the inner braid. Where the diameter change-over limit is such that Table IX or Table X and this section require different sizes of yarn on the inner and outer braids, both braids may be of the size of yarn as required on the outer braid.

TABLE XI.—BRAID.

Diameter Under Braid, *D*, in.	Size and Ply of Cotton Yarn, Minimum	No. of Ends	Thickness of Braid, Minimum, in.	Diameter, Mean Avg. (*D*+ *T*), in.	12-Carrier Braiders		16-Carrier Braiders		20-Carrier Braiders		24-Carrier Braiders		36-Carrier Braiders		48-Carrier Braiders	
					Picks per inch	Braid Angle, deg.	Picks per inch	Braid Angle, deg.	Picks per inch	Braid Angle, deg.	Picks per inch	Braid Angle, deg.	Picks per inch	Braid Angle, deg.	Picks per inch	Braid Angle, deg.
					STANDARD BRAID											
0.090	30/2	2	0.0163	0.106	27.7	57.0	22.6	43.0	....	....	....	....	....	....	....	....
0.091 to 0.110	30/2	2	0.0163	0.116	28.6	60.0	24.7	48.0	....	....	....	....	....	....	....	....
0.111 to 0.125	30/2	2	0.0163	0.134	29.8	65.0	27.0	55.0	23.0	44.0	....	....	....		....	....
0.126 to 0.150	30/2	2	0.0163	0.154	30.6	68.0	28.6	60.0	25.8	51.0	....		....		....	....
0.151 to 0.175	30/2	2	0.0163	0.179	31.2	71.0	29.8	64.0	27.8	58.0	....	....	....		....	....
0.176 to 0.200	30/2	2	0.0163	0.204	31.6	73.0	30.6	68.0	29.1	62.0	....		....		....	....
0.201 to 0.225	26/2	2	0.0175	0.231	30.2	74.3	29.3	69.4	28.1	63.9	26.6	58.1	....		....	....
0.226 to 0.250	26/2	2	0.0175	0.256	30.4	76.2	29.7	71.5	28.8	66.7	27.6	61.6	....		....	....
0.251 to 0.300	26/2	2	0.0175	0.293	30.4	78.0	30.2	74.0	29.5	70.0	28.6	65.0	....		....	....
0.301 to 0.350	26/2	2	0.0175	0.343	....	....	....	....	30.0	73.0	29.4	69.0	....		....	....
0.351 to 0.400	20/2	3	0.020	0.396	....	....	....	....	18.8	66.0	18.0	62.0	....		....	....
0.401 to 0.450	20/2	3	0.020	0.445	....		....	....	19.1	70.0	18.5	65.0	....		....	....
0.451 to 0.500	20/2	3	0.020	0.495	....	....	....	....	19.4	72.0	18.9	68.0	....		....	....
0.501 to 0.600	20/2	3	0.020	0.570	....	....	....	....	20.4	75.0	20.1	72.0	....		....	....
0.601 to 0.800	20/2	3	0.020	0.720	....	....	....	....	20.6	78.0	20.5	76.0	....		....	....
0.801 to 1.000	12/2	4	0.0258	0.925	....	....	....	....	11.8	73.7	11.6	70.3	10.7	59.8	9.2	48.6
1.001 to 1.200	12/2	4	0.0258	1.125	....	....	....	....	12.0	76.8	11.9	74.0	11.2	65.7	10.3	56.0
1.201 to 1.500	12/2	4	0.0258	1.376	....	....	....	....	....	....	12.0	77.0	11.6	70.2	11.0	63.2
1 501 to 2.000	8/2	5	0.0316	1.782	....	....	....	....	....	....	7.2	73.3	6.8	64.6	6.1	55.0
2.001 to 3.000	8/2	5	0.0316	2.532	....	....	....	....	....	....	7.3	78.2	7.1	72.2	6.9	66.2
					HEAVY BRAID											
0.090	20/2	2	0.020	0.110	22.6	52.5	16.5	35.5	....	....	....	....	....	....	....	....
0.091 to 0.110	20/2	2	0.020	0.120	23.6	56.0	18.9	41.7	....	....	....	....	....	....	....	....
0.111 to 0.125	20/2	2	0.020	0.138	24.6	58.3	21.5	49.3	....	....	....	....	....	....	....	....
0.126 to 0.150	20/2	2	0.020	0.158	25.7	64.6	23.3	55.3	20.0	44.8	....	....	....	....	....	....
0.151 to 0.175	20/2	2	0.020	0.183	26.4	67.5	24.8	60.5	22.7	52.5	19.2	42.5	....	....	....	....
0.176 to 0.200	20/2	2	0.020	0.208	26.9	70.4	25.5	65.8	23.7	57.0	21.6	49.5	....	....	....	....
0.201 to 0.225	16/2	2	0.0223	0.235	....	....	23.8	56.5	22.4	58.8	20.4	51.5	....	....	....	....
0.226 to 0.250	16/2	2	0.0223	0.260	....	....	25.8	77.8	23.0	62.0	21.6	55.6	....	....	....	....
0.251 to 0.300	16/2	3	0.0223	0.297	....	....	16.3	62.3	15.0	54.5	13.2	49.5	....	....	....	....
0.301 to 0.350	12/2	3	0.0258	0.351	....	....	....	....	13.2	55.4	11.8	47.3	....	....	....	....
0.351 to 0.400	12/2	3	0.0258	0.401	....	....	....	....	13.9	60.1	12.9	53.5	....	....	....	....
0.401 to 0.450	12/2	3	0.0258	0.451	....	....	....	....	14.4	63.9	13.6	58.1	....	....	....	....
0.451 to 0.500	12/2	3	0.0258	0.501	....	....	....	....	14.7	66.6	14.1	62.0	....	....	....	....
0.501 to 0.600	12/2	3	0.0258	0.576	....	....	....	....	....	....	15.2	66.4	13.3	55.7	....	....
0.601 to 0.800	8/2	3	0.0316	0.732	....	....	....	....	....	....	12.5	67.3	11.1	57.2	....	...
0.801 to 1.000	8/2	3	0.0316	0.932	....	....	....	....	....	....	12.9	72.3	12.0	65.0	10.8	53.0
1.001 to 1.200	6/2	2	0.037	1.137	....	....	....	....	....	....	....	....	17.0	74.9	16.4	67.7
1.201 to 1.500	6/2	2	0.037	1.387	....	....	....	....	....	....	....	....	17.2	77.7	16.8	71.8
1.501 to 2.000	4/2	3	0.0446	1.795	....	....	....	....	....	....	....	....	8.6	71.3	8.1	62.3
2.001 to 3.000	4/3	3	0.0556	2.556	....	....	....	....	....	....	....	....	6.6	72.8	6.3	64.5

(*f*) Where two braids are applied in one operation, these specifications shall apply to the inner and outer braids except that picks per inch and angle of braid for inner braid shall be governed by the construction of the machine.

(*g*) These specifications do not apply to braids for fixture wire or to fancy or special braids.

(*h*) In Table XI are tabulated the required picks per inch as calculated by the formula in Paragraph (*c*) and the corresponding braid angle for braids applied with the minimum size and ply of cotton, and a definite number of ends per carrier. However, it is not the intent of these specifications to limit the braids to the scope of Table XI since heavier cottons or a variation in ends per carrier are permissible providing all requirements of Sections 57 and 58 are fulfilled.

Measurements

59. The various quantities shall be determined as follows:

Diameter Under Braid, by a suitable micrometer reading.

Number of Carriers and *Ends per Carrier*, by examination of braid.

Picks per Inch, on a 4-in. sample after wiping off outside of braid with chloroform.

Angle of Braid by formula: Tangent of Angle $= \frac{2\pi P\ (D + T)}{C}$

Thickness of Braid (2 × diameter of single end of cotton), by measuring 25 per cent of the total number of threads with a paper micrometer having a foot 0.4 in. in diameter under a weight of 50 g. The sample of braid on which the average diameter is determined shall be prepared by immersing a 2-in. length of sample in chloroform in a suitable container for 5 minutes, decanting and repeating for 1 minute, and removing and drying it thoroughly by pressing it between cheese cloth.

Number of Tests

60. Samples shall be taken at the option of the purchaser.

(*E*) FINISH

Finish

61. The braid shall be completely saturated and finished with a black moisture-resistant compound. The surface of the wire shall be smooth and free from tackiness. The finish shall be capable of meeting the tests described in Sections 62 to 65, inclusive. Single conductor 600-v. wire for building purposes is required by the National Electrical Code to have a flame-proof finish. Where Underwriters jurisdiction applies the finish and tests shall be in accordance with Underwriters standards.

Bend Test

62. (*a*) *Sample.*—Each sample for the bend test shall be wrapped as specified in Paragraph (*b*) around a mandrel of the diameter given in the following table:

DIAMETER OF SAMPLE, IN.	RATIO OF MANDREL DIAMETER TO OUTSIDE DIAMETER OF WIRE OR CABLE
0 to 0.250	1½
0.251 to 0.375	2¼
0.376 to 0.500	3
0.501 to 0.750	4½
0.751 to 1.000	6
1.001 to 1.500	9
1.501 and up	12

(*b*) *Test Procedure.*—The samples shall be held at a temperature of 32 F. (0 C.) for approximately 1 hr. before bending, and upon removal from the cooling chamber shall be wrapped upon the mandrel at an approximately uniform rate not greater than 12 turns per minute. Single conductor No. 2 A.w.g. and smaller shall be wrapped three times around the mandrel; when requested single conductor wires larger than No. 2 A.w.g. and any multiple conductor shall be wrapped once around the mandrel.

(*c*) *Flaking and Cracking.*—The finishing material shall not flake off the wire or cable during the bend test or as a result of the bend test without actually rubbing the surface of the wire. Cracking of the finish will not be cause for rejection.

Melt Test

63. A piece of clean white glazed paper shall be wrapped around a 6-in. sample of finished wire or cable and this shall be suspended horizontally in an electrically heated oven, equipped with suitable automatic temperature control, which has been heated to 125 F. (52 C.) and the wire or cable shall be maintained at this temperature for 30 min. The wire or cable with the paper wrapping shall be cooled before inspection. The wire shall be rejected if the compound becomes sufficiently fluid to be transferred to the paper or form a ridge perceptible to the finger upon it.

Drip Test

64. Samples for the drip test shall be cut in lengths of 6 in. and the wire bared for a distance of ¾ in. from each end. The sample with one end secured in a suitable clamp shall then be suspended at an angle of 45 deg. in an electrically heated oven, equipped with suitable automatic temperature control, which has been heated to 180 F. (82.2 C.) and the wire or cable shall be maintained at this temperature for 1 hr. At the end of this time the compound shall not have dripped, flowed onto the surface of the lower bared end of the conductor, formed globules on the lower side of the sample or bubbles upon its surface.

Saturating and Finishing Compounds

65. The saturating and finishing compounds shall have no injurious effect upon the rubber insulation or the coverings during application.

(*F*) LEAD SHEATHS

Composition

66. (*a*) Lead sheaths shall consist of Grade II Chemical Lead or Grade III Common Lead not less than 99.85 per cent pure as specified in the Standard Specifications for Pig Lead (A.S.T.M. Designation: B 29) of the American Society for Testing Materials.[1] It shall be obtained from properly branded pig lead.

(*b*) Lead sheaths shall be tightly formed around the core of the wire or cable.

Thickness

67. Lead sheaths shall have an average thickness not less than that indicated in Table XII and the minimum thickness shall in no place be less than 90 per cent of the required average thickness.

TABLE XII.—THICKNESS OF LEAD SHEATHS.

DIAMETER OF CORE, IN.[a]	THICKNESS OF SHEATH IN SIXTY-FOURTHS OF AN INCH
Over 3.000	9
2.001 to 3.000	8
1.501 to 2.000	7
1.051 to 1.500	6
0.701 to 1.050	5
0.426 to 0.700	4
0.000 to 0.425	3

[a] For twin cables, use the major axis.

Measurement

68. A short section of the lead sheath shall be removed from the wire or cable and the thickness measured with a suitable micrometer caliper at not less than ten points, approximately equally spaced, care being taken to include the thinnest and the thickest parts.

(*G*) INSPECTION AND REJECTION

Inspection and Test

69. (*a*) Inspection shall be made prior to shipment and at the place of manufacture.

(*b*) The manufacturer shall notify the purchaser sufficiently in advance of the completion of the wire or cable to permit of arrangement of inspection.

(*c*) The inspector representing the purchaser shall have free entry at all times, while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the wire ordered, except compounding room. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the wire is being furnished in accordance with these specifications.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 678.

(*d*) The purchaser at his option may make the various tests on samples in his own laboratory or elsewhere, but such tests shall be made at the expense of the purchaser.

Rejection

70. Each coil, reel or length which fails to comply with the physical and electrical requirements of these specifications shall be rejected.

Rehearing

71. Samples of rejected material shall be preserved for two weeks from date of test report. In case of dissatisfaction with the results of test, the manufacturer may make claim for a rehearing within that time. Sealed duplicate samples may be retained by the manufacturer at his option.

APPENDIX I

ANNEALED COPPER STANDARD[1]

The following shall be taken as normal values for standard annealed copper:

(*a*) *Resistance.*—At a temperature of 20° C. the resistance of a wire of standard annealed copper 1 meter in length and of a uniform section of 1 sq. mm. is $\frac{1}{58}$ ohm = 0.017241 ohm.

(*b*) *Density.*—At a temperature of 20° C. the density of standard annealed copper is 8.89 g. per cu. cm.

(*c*) *Temperature Coefficient of Resistance.*—At a temperature of 20° C. the "constant mass" temperature coefficient of resistance of standard annealed copper, measured between two potential points rigidly fixed to the wire, is 0.00393 = 1/254.45 per degree Centigrade.

(*d*) Other values of resistivity of standard annealed copper at 20° C. deduced from Paragraphs (*a*) and (*b*) are as follows:

Resistance of a wire of uniform section, 1 meter long and weighing 1 g. = 0.15328 ohm.

Resistance of a wire with a uniform section of 1 cir. mil and 1 ft. long = 10.371 ohm.

Resistance between opposite faces of 1 cu. cm. = 1.7241 microhm.

Resistance between opposite faces of 1 cu. in. = 0.67879 microhm.

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[1] Extracts from American Institute of Electrical Engineers' Standard No. 30 (1932).

APPENDIX II

Interpretation of Insulation Thicknesses of Table IV

For series-street-lighting cable, use the following procedure:

A1. (*a*) If the transformer is not equipped with protectors, use the open circuit secondary voltage of the transformer as rated voltage of the cable and select the proper wall thickness from Table IV.

(*b*) If the transformer is equipped with protectors, use the closed or loaded secondary voltage of the transformer as the rated circuit voltage of the cable and select the proper wall thickness from Table IV.

Note.—It is preferable, both in Paragraphs (*a*) and (*b*) above, to select a cable with a higher voltage rating than obtained using the above procedure, both as a measure of safety and to allow the substitution of a larger transformer without necessitating the replacement of the cable.

The thickness of insulation for the various systems shall be determined as follows:

A2. (*a*) For three-phase systems with grounded or ungrounded neutral, use thickness values in accordance with Table IV.

(*b*) For single or two-phase systems up to and including 5000 v., use thickness values in accordance with Table IV, as specified for grounded neutral. Where it is not definitely specified that a line operates as an isolated single or two-phase system it shall be considered as a branch of a grounded three-phase circuit and the thickness of insulation shall be that required by the phase-to-phase voltage of this grounded, three-phase circuit as given in Table IV.

(*c*) For single or two-phase systems operating at over 5000 v. with one side grounded, multiply the circuit voltage (phase-to-phase) by 1.73 and use the resulting voltage value to select the corresponding insulation thickness for grounded neutral as specified in Table IV.

(*d*) For single or two-phase systems operating at over 5000 v. with the center grounded, multiply the circuit voltage (phase-to-phase) by 0.866 ($\frac{1}{2}\sqrt{3}$) and use the resulting voltage value to select the corresponding insulation thickness for grounded neutral as specified in Table IV.

(*e*) For single or two-phase ungrounded systems operating at over 5000 v., multiply the circuit voltage (phase-to-phase) by 0.866 ($\frac{1}{2}\sqrt{3}$) and use the resulting voltage value to select the corresponding insulation thickness for ungrounded neutral as specified in Table IV.

(*f*) For direct-current systems, consider them the same as single-phase alternating-current systems in accordance with Paragraph A2 (*b*).

TENTATIVE SPECIFICATIONS
FOR
INSULATED WIRE AND CABLE: HEAT-RESISTING RUBBER COMPOUND[1]

A.S.T.M. Designation: D 469 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.[2]

Scope

1. (*a*) These specifications cover wire and cable insulated with a vulcanized rubber compound. The suitability of the compound as insulation shall be determined solely by physical and electrical tests. The compound shall be called heat-resisting rubber compound. This compound is ordinarily recommended for use at conductor temperatures not in excess of 75 C.

(*b*) Except for the rubber insulation and methods of testing, wire and cable supplied under these specifications shall conform to the Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (A.S.T.M. Designation: D 27 – 37 T) of the American Society for Testing Materials.[3]

SPECIFICATIONS

INSULATION

Workmanship

2. (*a*) The insulation shall be homogeneous in character, tough, elastic and applied concentrically about the conductor and shall fit tightly thereto. Where the insulation is applied in more than one layer, adjacent layers shall be vulcanized into a homogeneous mass.

(*b*) Where repairs or joints are made in the insulation, the work shall be done in such manner that the repaired part of the joint, and all parts affected in the process, shall be as strong and durable electrically and mechanically as the remainder of the insulation and shall not exceed the limitations on the thickness specified in Section 7.

Shielding

3. (*a*) Braided cables not supported on insulators when operating at 6001 v. and over with the neutral grounded, or 3501 v. and over with the

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

[2] Accepted for publication as tentative by Committee E-10 on Standards, August 26, 1937.

[3] See p. 1070.

neutral ungrounded shall be shielded or suitably protected against corona. For special installations, such as braided cables connected to an overhead line, shielding may be provided at voltages lower than those given herein.

(*b*) All lead-covered multiple conductor cables operating at 6001 v. and over with the neutral ungrounded shall be shielded or suitably protected against corona.

(*c*) The shielding shall consist of a tinned copper tape at least 3 mils thick or braid of fine tinned copper wires.

Tape and Braid Coverings

4. The insulation of braided wires up to No 7 A.w.g., inclusive, shall be covered with a single cotton braid. On larger sizes the fibrous covering shall consist of a cable tape and cotton braid or two cotton braids. The cable tape may be applied before vulcanization. In multiple conductor cables the individual conductors shall not be braid-covered unless specified.

MANUFACTURE

Composition

5. The insulation shall consist of a properly vulcanized rubber compound that will meet the test requirements prescribed in these specifications.

PHYSICAL PROPERTIES

Physical Properties

6. The insulation shall conform to the following requirements as to physical properties:

	MAXIMUM	MINIMUM
Tensile strength, lb. per sq. in.	....	1500
Tensile stress at 200 per cent elongation, lb. per sq. in.	....	400
Elongation at rupture, per cent	....	400
Set in 2-in. gage length, in.	$\frac{3}{8}$	....
Tensile strength after air pressure heat test, lb. per sq. in.	....	1000
Depreciation in tensile strength after air pressure heat test, per cent	50	....
Elongation at rupture after air pressure heat test, per cent	....	300
Depreciation in elongation after air pressure heat test, per cent	35	....
Tensile strength after air oven test, lb. per sq. in.	....	1000
Depreciation in tensile strength after air oven test, per cent	50	....
Elongation at rupture after air oven test, per cent	....	300
Depreciation in elongation after air oven test, per cent	35	....
Depreciation in tensile strength and elongation after oxygen pressure test, per cent	25	....

THICKNESS

Thickness

7. (*a*) The average thickness of the insulation shall be not less than that given in Table I. The minimum thickness shall be not less than 95 per cent of the thickness required by Table I for insulations less than $\frac{4}{64}$ in. in thickness and not less than 90 per cent of the specified thickness for insulations of $\frac{4}{64}$ in. and greater.

TENTATIVE SPECIFICATIONS

FOR

INSULATED WIRE AND CABLE: HEAT-RESISTING RUBBER COMPOUND[1]

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(*b*) All lead-covered multiple conductor cables operating at 6001 v. and over with the neutral ungrounded shall be shielded or suitably protected against corona.

(*c*) The shielding shall consist of a tinned copper tape at least 3 mils thick or braid of fine tinned copper wires.

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Elongation at rupture, per cent	....	400
Set in 2-in. gage length, in.	$\frac{3}{8}$	....
Tensile strength after air pressure heat test, lb. per sq. in.	....	1000
Depreciation in tensile strength after air pressure heat test, per cent	50	....
Elongation at rupture after air pressure heat test, per cent	....	300
Depreciation in elongation after air pressure heat test, per cent	35	....
Tensile strength after air oven test, lb. per sq. in.	....	1000
Depreciation in tensile strength after air oven test, per cent	50	....
Elongation at rupture after air oven test, per cent	....	300
Depreciation in elongation after air oven test, per cent	35	....
Depreciation in tensile strength and elongation after oxygen pressure test, per cent	25	....

THICKNESS

Thickness

7. (*a*) The average thickness of the insulation shall be not less than that given in Table I. The minimum thickness shall be not less than 95 per cent of the thickness required by Table I for insulations less than $\frac{4}{64}$ in. in thickness and not less than 90 per cent of the specified thickness for insulations of $\frac{4}{64}$ in. and greater.

TABLE I.—THICKNESS OF INSULATION AND ALTERNATING TEST VOLTAGE.

NOTE 1.—Rated circuit voltage of alternating-current circuits is the phase-to-phase voltage.

NOTE 2.—For an intermediate size of wire or an intermediate rated circuit voltage, the thickness and test voltage shall be that for the next larger size or higher rated circuit voltage.

NOTE 3.—For non-leaded submarine cables, $\frac{1}{32}$ in. should be added to the specified respective walls of insulation in this table for all sizes and voltages.

NOTE 4.—Rated circuit voltages from 0 to 5000 v. in this table apply to wire for use on grounded and ungrounded circuits.

NOTE 5.—Above 5000 v. alternating-current, special high-voltage rubber compounds are preferred according to manufacturer's recommendations.

Rated Circuit Voltage	Size of Conductor, A. w. g. Numbers or Circular Mils	Insulation Thickness on each Conductor, sixty-fourths of an inch	Test Voltage
0 to 300	No. 18 to No. 16	2	1 000
0 to 600	No. 14 to No. 9	3	3 000
	No. 8 to No. 2	4	3 500
	No. 1 to No. 0000	5	4 000
	225 000 to 500 000 cir. mils	6	5 000
	525 000 to 1 000 000 cir. mils	7	6 000
	Over 1 000 000 cir. mils	8	7 000
601 to 1000	No. 14 to No. 8	4	5 000
	No. 7 to No. 2	5	6 000
	No. 1 to No. 0000	6	7 500
	225 000 to 500 000 cir. mils	7	9 000
	525 000 to 1 000 000 cir. mils	8	10 000
	Over 1 000 000 cir. mils	9	11 000
1001 to 2000	No. 14 to No. 8	5	6 000
	No. 7 to No. 2	6	7 500
	No. 1 to No. 0000	7	9 000
	225 000 to 500 000 cir. mils	8	10 000
	525 000 to 1 000 000 cir. mils	9	11 000
	Over 1 000 000 cir. mils	9	11 000
2001 to 3000	No. 14 to No. 8	7	9 000
	No. 7 to No. 2	8	10 000
	No. 1 to No. 0000	8	10 000
	225 000 to 500 000 cir. mils	9	11 000
	525 000 to 1 000 000 cir. mils	9	11 000
	Over 1 000 000 cir. mils	10	12 500
3001 to 4000	No. 14 to No. 8	9	11 000
	No. 7 to No. 2	9	11 000
	No. 1 to No. 0000	9	11 000
	225 000 to 500 000 cir. mils	10	12 500
	525 000 to 1 000 000 cir. mils	10	12 500
	Over 1 000 000 cir. mils	11	13 500
4001 to 5000	No. 14 to No. 8	10	12 500
	No. 7 to No. 2	10	12 500
	No. 1 to No. 0000	10	12 500
	225 000 to 500 000 cir. mils	11	13 500
	525 000 to 1 000 000 cir. mils	11	13 500
	Over 1 000 000 cir. mils	12	15 000
5001 to 6000 (grounded)	No. 14 to No. 0000	10	12 500
	225 000 to 1 000 000 cir. mils	11	13 500
	Over 1 000 000 cir. mils	12	15 000
5001 to 6000 (ungrounded)	No. 14 to No. 0000	12	15 000
	225 000 to 1 000 000 cir. mils	12	15 000
	Over 1 000 000 cir. mils	13	16 500
6001 to 7000 (grounded)	No. 8 to No. 0000	11	13 500
	225 000 to 1 000 000 cir. mils	11	13 500
	Over 1 000 000 cir. mils	12	15 000
6001 to 7000 (ungrounded)	No. 8 to No. 0000	14	17 500
	225 000 to 1 000 000 cir. mils	14	17 500
	Over 1 000 000 cir. mils	15	19 000
7001 to 8000 (grounded)	No. 8 to No. 0000	12	15 000
	225 000 to 1 000 000 cir. mils	12	15 000
	Over 1 000 000 cir. mils	13	16 500
7001 to 8000 (ungrounded)	No. 8 to No. 0000	16	20 000
	225 000 to 1 000 000 cir. mils	16	20 000
	Over 1 000 000 cir. mils	17	21 000

(*b*) The thicknesses in Table I apply to single conductor cable and the individual conductors of multiple conductor cables, leaded or braided, except special applications such as aerial, non-leaded submarine, and vertical riser cable.

NOTE.—In the Appendix will be found an interpretation of the insulation thicknesses given in Table I.

ELECTRICAL PROPERTIES

High Voltage Test

8. Each coil, reel, or length of wire or cable after vulcanization shall successfully withstand the application of an alternating voltage of not less than that prescribed in Table I for a period of 5 min. (See Sections 33 and 34.)

Insulation Resistance

9. Each coil, reel, or length of wire or cable after being subjected to the high voltage test specified in Section 8 shall have an insulation resistance not less than that given in Table II.

Moisture Absorption

10. (*a*) The increase in the specific inductive capacity of the insulation shall be used as an indication of the amount of moisture absorbed.

(*b*) The specific inductive capacity after 24-hr. immersion in water shall not exceed five. The increase between the ends of the first and the fourteenth days shall not exceed 20 per cent and that between the ends of the seventh and fourteenth days, 5 per cent.

(*c*) The specific inductive capacity shall be determined at either 1000 or 60 cycles. Where measured at 1000 cycles, the voltage impressed upon the conductor shall not exceed 10 v. Where measured at 60 cycles, the voltage impressed upon the conductor shall be equivalent to a stress of between 30 to 40 v. per mil of insulation.

(*d*) The measurements shall be made as described in Section 37.

METHODS OF TESTING

TENSILE STRENGTH, TENSILE STRESS, ELONGATION AT RUPTURE, AND SET

Sampling for Physical Tests

11. (*a*) Wire up to and including 250,000 cir. mils in size shall be sampled in accordance with the following requirements:

QUANTITY OF WIRE	NUMBER OF SAMPLES
2000 ft. and under	none
2001 to 10,000 ft.	1 for each 2000 ft. or fraction thereof
10,001 to 100,000 ft.	5 plus 1 for each 10,000 ft. additional over 10,000 ft.
100,001 to 500,000 ft.	14 plus 1 for each 25,000 ft. over 100,000 ft.

(*b*) For wire larger than 250,000 cir. mils, one sample shall be selected from quantities between 1000 and 2000 ft. and for larger quantities double those specified in Paragraph (*a*).

TABLE II.—INSULATION RESISTANCE, MEGOHMS—1000 FT. AT 60 F. (15.5 C.).

NOTE 1.—This table is based on a constant $K = 21{,}120$ in the following formula:

$R = K \log_{10}\frac{D}{d}$; where R = resistance in megohms—1000 ft., K = constant.

D = diameter over insulation, on each conductor, and d = diameter over conductor.

NOTE 2.—For an intermediate size, the insulation resistance shall be that of the next larger size.

Size of Conductor, A.w.g. Numbers or Circular Mils	Solid or Stranded	Diameter, in.	Insulation Thickness in Sixty-fourths of an Inch									
			2	3	4	5	6	7	8	9	10	12
No. 18	Solid	0.040	8600	11 100	....	....	....	....	....	....	....	....
No. 16	Solid	0.051	7300	9 500	....	....	....	....	....	....	....	....
No. 14	Solid	0.064	....	8 300	9 900	11 300	12 550	13 650	14 600	15 450	16 250	17 650
No. 12	Solid	0.081	....	7 050	8 550	9 850	11 000	12 000	12 900	13 750	14 500	15 850
No. 10	Solid	0.102	....	6 000	7 350	8 500	9 600	10 500	11 350	12 100	12 850	14 150
No. 8	Solid	0.128	....	....	6 200	7 300	8 300	9 150	9 950	10 650	11 350	12 550
No. 6	Stranded	0.184	....	....	4 750	5 600	6 400	7 200	7 900	8 500	9 100	10 200
No. 4	Stranded	0.232	....	....	3 900	4 700	5 450	6 100	6 700	7 250	7 800	8 850
No. 2	Stranded	0.292	....	....	3 250	3 900	4 550	5 150	5 650	6 150	6 650	7 600
No. 1	Stranded	0.332	....	....	....	3 550	4 100	4 650	5 150	5 600	6 100	6 950
No. 0	Stranded	0.373	....	....	....	3 200	3 750	4 200	4 700	5 150	5 550	6 350
No. 00	Stranded	0.418	....	....	....	2 900	3 400	3 850	4 300	4 700	5 100	5 850
No. 000	Stranded	0.470	....	....	....	2 600	3 100	3 500	3 900	4 250	4 650	5 400
No. 0000	Stranded	0.528	....	....	....	2 400	2 800	3 150	3 550	3 900	4 200	4 900
250 000 cir. mils	Stranded	0.575	....	....	....	....	2 600	2 950	3 300	3 650	4 000	4 600
350 000 cir. mils	Stranded	0.681	....	....	....	....	2 250	2 550	2 850	3 150	3 450	4 000
500 000 cir. mils	Stranded	0.814	....	....	....	....	1 900	2 200	2 450	2 700	2 950	3 450
750 000 cir. mils	Stranded	0.998	....	....	....	....	....	1 800	2 050	2 250	2 500	2 900
1 000 000 cir. mils	Stranded	1.152	....	....	....	....	....	1 600	1 800	2 000	2 200	2 600
1 250 000 cir. mils	Stranded	1.289	....	....	....	....	....	....	1 600	1 800	2 000	2 350
1 500 000 cir. mils	Stranded	1.412	....	....	....	....	....	....	1 500	1 650	1 850	2 150
1 750 000 cir. mils	Stranded	1.526	....	....	....	....	....	....	1 400	1 550	1 700	2 000
2 000 000 cir. mils	Stranded	1.631	....	....	....	....	....	....	1 300	1 500	1 600	1 900

Size of Conductor, A.w.g. Numbers or Circular Mils	Solid or Stranded	Diameter, in.	Insulation Thickness in Sixty-fourths of an Inch				
			13	14	15	16	17
No. 8	Solid	0.128	13 100	13 600	14 100	14 600	14 900
No. 6	Stranded	0.184	10 650	11 150	11 500	12 000	12 450
No. 4	Stranded	0.232	9 300	9 700	10 100	10 500	10 900
No. 2	Stranded	0.292	7 950	8 400	8 800	9 150	9 500
No. 1	Stranded	0.332	7 350	7 650	8 000	8 400	8 750
No. 0	Stranded	0.373	6 750	7 050	7 400	7 800	8 100
No. 00	Stranded	0.418	6 200	6 500	6 900	7 150	7 500
No. 000	Stranded	0.470	5 700	6 150	6 350	6 650	6 900
No. 0000	Stranded	0.528	5 200	5 500	5 800	6 050	6 350
250 000 cir. mils	Stranded	0.575	4 900	5 200	5 450	5 750	5 950
350 000 cir. mils	Stranded	0.681	4 250	4 500	4 700	4 950	5 200
500 000 cir. mils	Stranded	0.814	3 700	3 950	4 150	4 350	4 550
750 000 cir. mils	Stranded	0.998	3 100	3 300	3 500	3 700	3 900
1 000 000 cir. mils	Stranded	1.152	2 750	2 950	3 100	3 300	3 450
1 250 000 cir. mils	Stranded	1.289	2 500	2 650	2 850	3 000	3 150
1 500 000 cir. mils	Stranded	1.412	2 300	2 450	2 600	2 700	2 950
1 750 000 cir. mils	Stranded	1.526	2 150	2 300	2 450	2 600	2 750
2 000 000 cir. mils	Stranded	1.631	2 050	2 200	2 300	2 450	2 600

Test Specimens

12. (*a*) *Size.*—The test specimen may be the entire section of the insulation in the case of small wires, but in the case of wire and cable of

No. 6 A.w.g. and larger or with an insulation thickness greater than $\frac{3}{32}$ in., samples approximately square in section shall be cut from the insulation with a cross-section not greater than 0.025 sq. in. The test specimen shall be as free as possible from surface incisions and imperfections.

(*b*) *Condition and Age.*—Samples of the insulated wire or cable for physical and accelerated aging tests shall be taken after vulcanization and prior to the application of any covering except that a cable tape may be applied before vulcanizing. No tests shall be made within 24 hr. nor later than 60 days after vulcanization unless agreed to by the manufacturer. Specimens shall not be heated, immersed in water or subjected to any mechanical or chemical treatment not specifically prescribed in these specifications. Specimens for accelerated aging tests having cable tape applied prior to vulcanization shall be aged with such tape removed.

(*c*) *Buffing.*—In the event of any irregularities on the surface of the test specimen including corrugations from large strands, it shall be made smooth and of uniform thickness by buffing.

Calculation of Area

13. Calculation of the area of the test specimens shall be made as follows:

(*a*) Where the total cross-section of the insulation is used, the area shall be taken as the difference between the area of the circle whose diameter is the average outside diameter of the insulation and the area of the conductor. The area of the stranded conductor shall be calculated from its maximum diameter.

(*b*) Where a segment or sector of the insulation has to be taken in the case of a large wire or cable or where the insulation is thin, the area shall be calculated as the thickness times the width. (This applies either to a straight test specimen or one stamped out with a die, and assumes that corrugations have been removed by buffing.)

Temperature at Test

14. Physical tests shall be made at a room temperature not less than 65 F. (18.3 C.) nor more than 90 F. (32.2 C.) and the test specimen shall have been kept at the room temperature not less than 30 min. prior to the test.

Tension Testing Machine

15. The tension testing machine shall be power-driven and preferably of the pendulum type. The machine shall be accurate within 1 per cent of the breaking load. A spring balance type of apparatus may be used if provided with a device which will indicate the actual maximum load at which rupture takes place and if provision is made to prevent recoil of the spring.

Tensile Strength Test

16. The tensile strength test shall be made on a specimen which has not been previously stretched. The specimen shall have a length of not less than 6 in., and shall be marked with gage marks 2 in. apart and placed in the jaws of the testing machine with a maximum distance between jaws of 4 in. The specimen shall be stretched at the rate of 20 in. per min. (jaw speed) until it breaks. The test specimen shall break between the gage marks. The tensile strength shall be calculated upon the area of the original specimen.

Tensile Stress Test

17. The tensile stress test shall be made simultaneously with the tensile strength test by recording the load when the 2-in. gage length marks are 6 in. apart, that is, at 200 per cent elongation. The tensile stress shall be calculated upon the area of the original specimen.

Elongation at Rupture

18. Elongation at rupture shall be determined simultaneously with the tensile strength test by reading the distance in inches between gage marks at the breaking point. The elongation at rupture shall be taken as the distance between gage marks at rupture less 2 in. (the original gage length). The percentage of elongation at rupture is the elongation in inches divided by the original gage length (2 in.) and multiplied by 100 to express as a percentage.

Set Test

19. The set test shall be made on a separate test specimen having a length of not less than 6 in. and marked with gage marks 2 in. apart. The specimen shall be placed in the jaws of the testing machine with a maximum distance between jaws of 4 in. and shall be stretched at the rate of 20 in. per min. (jaw speed) until the gage marks are 6 in. apart. The test specimen shall then be released within 5 sec. and the distance between gage marks shall be determined 1 min. after the beginning of release. The set is the difference between this length and the original 2-in. gage length.

Retests, Physical Properties

20. Where tested in accordance with Sections 16, 17, 18, and 19, if a specimen fails to conform to any one of the requirements for tensile strength, tensile stress, elongation at rupture, or set specified in Section 6, two additional specimens shall be taken from the same sample and tested, and the average of the results shall determine whether the material conforms to the requirements.

AGING TESTS

Samples for Aging Tests

21. For aging tests the wire shall be sampled as follows:

No sample shall be selected where the quantity is less than 2000 ft. of wire or cable.

For sizes less than 250,000 cir. mils, one sample shall be selected for each quantity ordered between 2000 ft. and 50,000 ft. of wire or cable and one additional sample for each additional 50,000 ft. thereafter.

For sizes 250,000 cir. mils and over, one sample shall be selected for each quantity ordered between 2000 ft. and 25,000 ft. of wire or cable and one additional sample for each additional 25,000 ft. thereafter.

Test Specimens

22. Twelve test specimens of similar size and shape shall be prepared from each sample selected in accordance with Section 21, three for the determination of the initial or unaged properties, three for the air pressure heat test, three for the air oven test and three for the oxygen pressure test. One specimen of each three shall be tested and the other two held as spares except that where only one sample is selected all three specimens shall be tested and the average of the results reported.

Aging Test Conditions

23. (*a*) In the case of wire and cable smaller than No. 6 A.w.g. with an insulation thickness less than $\frac{3}{32}$ in., the insulation shall be heated in the air pressure heat test apparatus, in the air oven and in the oxygen pressure test apparatus without removing the conductor.

(*b*) In the case of wire and cable of No. 6 A.w.g. and larger or with an insulation thickness greater than $\frac{3}{32}$ in., samples approximately square in section shall be cut from the insulation with a cross-section not greater than 0.025 sq. in.

(*c*) Die-cut samples shall be buffed before being subjected to the accelerated aging test.

(*d*) The test specimens shall have no protective covering and shall be suspended vertically in such a manner that they will not come in contact with each other or with the sides of the pressure vessel or oven.

(*e*) Physical tests on both the aged and unaged specimens shall be made at the same time. The aged specimens shall have a rest period of not less than 16 hr. nor more than 48 hr. between the completion of the aging test and the determination of the physical properties.

(*f*) Should the first set of specimens selected under Section 20 fail to meet the specifications, two other sets of specimens shall be tested and if either of these sets fails, the coils, reels or lengths will be rejected.

Air Pressure Heat Test

24. The test specimens shall be heated in air at a pressure of 80 lb. per sq. in. at a temperature of from 259 to 261 F. for a period of 20 hr. The air pressure heat test shall be conducted in a steam-jacketed vessel or other

suitably and uniformly heated chamber. The air pressure heat test apparatus shall be so regulated that the temperature inside the chamber shall reach the test temperature within 15 min. after inserting the test specimens. The air used shall be substantially free from oil and moisture. The pressure shall be reduced at a uniform rate, requiring at least 2 min., when specimens are removed. The temperature shall be recorded automatically on a chart.

Air Oven Test

25. The test specimens shall be heated at a temperature of from 249 to 251 F. for a period of 120 hr. in an oven having a free circulation of fresh air. The oven temperature shall be recorded automatically on a chart. Except as otherwise specified in these specifications, the oxygen pressure test shall be made in accordance with Tentative Methods of Test for Accelerated Aging of Vulcanized Rubber (A.S.T.M. Designation: D 428 - 36 T) of the American Society for Testing Materials.[1]

Oxygen Pressure Test

26. The test specimens shall be heated in an atmosphere of oxygen at a pressure of 300 lb. per sq. in. at a temperature of from 157 to 159 F. for a period of 504 hr. The weight of the rubber in the vessel shall not be over 2 g. per cu. in. of space in the vessel. The pressure shall be reduced at a uniform rate, requiring at least 2 min., when specimens are removed. The temperature shall be recorded automatically on a chart. The oxygen pressure test shall be made only when requested by the purchaser. Except as otherwise specified in these specifications, the oxygen pressure test shall be made in accordance with Tentative Methods of Test for Accelerated Aging of Vulcanized Rubber (A.S.T.M. Designation: D 428 – 36 T) of the American Society for Testing Materials.[1]

Rejection

27. Where ten or more samples are selected in any inspection lot, all coils, reels or lengths shall be rejected if more than 10 per cent of the samples fail. If 10 per cent or less fail, each coil, reel or length may be tested and shall be accepted or rejected upon the results of such individual tests. Where the number of samples selected in any inspection lot is less than ten, all coils, reels, or lengths shall be rejected if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel or length may be tested and shall be accepted or rejected upon the results of such individual tests.

THICKNESS MEASUREMENTS

Apparatus

28. The thickness measurements may be made with any type of micrometer reading to 0.001 in., suitable for measurements of this character.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1113 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1274.

Number of Measurements

29. Where the lot of wire to be inspected consists of two coils or reels or less, at least one determination of the thickness shall be made on each coil or reel. Where the lot consists of more than two coils or reels and less than 20 coils or reels, at least one determination of the thickness shall be made on each of two coils or reels taken at random. If the lot consists of 20 or more coils or reels, not less than 10 per cent of the coils or reels shall be selected at random and at least one determination of the thickness made on each coil or reel so selected.

Procedure

30. The average thickness of the insulation shall be taken as one-half the difference between the mean of the maximum and minimum diameters measured at any point and the average diameter of the conductor measured at the same point. The minimum thickness shall be taken as the difference between a measurement made over the conductor plus the thinnest wall and the diameter of the conductor. (The first measurement is made by "slicing off" the heavier side of the insulation.)

In the case of multiple conductor cable, the measurements shall be made on the individual wires before being cabled.

Rejection

31. If the thickness of the insulation of any coil or reel is found to be less than the specified value, that coil or reel shall be rejected and a thickness measurement on each of the remaining coils or reels shall be made.

ELECTRICAL TESTS

Place

32. Electrical tests of wire and cable shall be made at the place of manufacture.

High Voltage Test

33. The high voltage test shall be made with alternating potential from a transformer and generator of ample capacity and in no case less than 5 kva. The frequency of the test voltage shall be not greater than 100 cycles and shall have a wave shape approximating as closely as possible a sine curve.

Rate of Application

34. The initally applied voltage shall be not greater than the rated voltage and the rate of increase shall be approximately uniform and not over 100 per cent in 10 sec. nor less than 100 per cent in 60 sec.

High Voltage Testing

35. (*a*) *General.*—Where insulated conductors are subjected to a high voltage test in water they shall be immersed in a grounded water bath and tested while still immersed.

(*b*) *Single Conductor.*—Single conductors shall be tested after 12-hr. immersion and without covering except a tape or braid applied before vulcanization.

Rubber sheathed conductors shall be immersed and tested after the sheath is applied.

Where a metallic sheath is applied, the conductors shall be retested against the sheath.

Single conductor cables shall be tested between conductor and sheath or water in which they are immersed at the test voltage required by Table I.

(*c*) *Multiple Conductor.*—The individual conductors of multiple conductor cables shall be tested before assembling as prescribed in Paragraph (*b*).

TABLE III.—TEMPERATURE COEFFICIENTS.

Temperature		Temperature Coefficient	Temperature		Temperature Coefficient
Deg. Fahrenheit	Deg. Centigrade		Deg. Fahrenheit	Deg. Centigrade	
46	7.8	0.65	61	16.1	1.03
47	8.3	0.67	62	16.7	1.07
48	8.9	0.69	63	17.2	1.10
49	9.4	0.71	64	17.8	1.13
50	10.0	0.73	65	18.3	1.17
51	10.6	0.76	66	18.9	1.20
52	11.1	0.78	67	19.4	1.24
53	11.7	0.80	68	20.0	1.28
54	12.2	0.83	69	20.6	1.32
55	12.8	0.86	70	21.1	1.36
56	13.3	0.88	71	21.7	1.40
57	13.9	0.91	72	22.2	1.45
58	14.4	0.94	73	22.8	1.50
59	15.0	0.97	74	23.3	1.55
60	15.6	1.00	75	23.9	1.59

After assembly, each conductor shall be tested against the adjacent conductors. Where a metallic sheath is applied, each conductor shall in addition be tested against the sheath.

Cables having a rubber or other non-metallic waterproof sheath shall be immersed for 12 hr. and the conductors tested against ground. Cables covered with cable tape and braid or double braid with a weather-resistant finish shall not be immersed for testing.

Multiple conductor cables after assembly shall be tested at the test voltage required by Table I.

Insulation Resistance

36. (*a*) The insulation resistance shall be measured after the high voltage test has been made and while the wire or cable is still immersed in water. The measurement shall be made after 1-min. electrification with a continuous e.m.f. of from 100 to 500 v., the conductor being maintained negative to the water. The temperature of the water shall be within the limits given in Table III.

Temperature Coefficient

(*b*) If the temperature at which the insulation resistance was measured differs from 60 F. (15.6 C.), the resistance shall be reduced to that at 60 F. (15.6 C.) by multiplying the measured value by the coefficient in Table III corresponding to the temperature at which the measurement was made.

Moisture Absorption Test

37. (*a*) A 15-ft. sample of the insulated wire or cable shall be taken after vulcanization and prior to the application of any coverings except tape applied before vulcanizing. Such cable tape shall be removed before making the moisture absorption test. The middle 10 ft. of the sample shall be immersed in distilled water for a period of 14 days with the $2\frac{1}{2}$-ft. portion at each end kept above the water as leakage insulation. The three measurements specified in Section 10 shall be taken with the water at the same temperature. The water shall be maintained at room temperature but not less than 70 F. (21 C.).

(*b*) The specific inductive capacity of the insulation shall be determined at commercial frequencies and shall be calculated as follows:

$$\text{Specific inductive capacity} = 13{,}600\, C \log_{10} \frac{D}{d}$$

where C = the capacity in microfarads of 10 ft. of sample,

D = the diameter over insulation, and

d = the diameter over conductor.

The moisture absorption test shall be made only when requested by the purchaser.

Rejection

38. Each coil, reel or length which fails to conform to the electrical requirements of these specifications shall be rejected.

APPENDIX

INTEPRETATION OF INSULATION THICKNESSES OF TABLE I

For series-street-lighting cable, use the following procedure:

A1. (*a*) If the transformer is not equipped with protectors, use the open circuit secondary voltage of the transformer as rated voltage of the cable and select the proper wall thickness from Table I.

(*b*) If the transformer is equipped with protectors, use the closed or loaded secondary voltage of the transformer as the rated circuit voltage of the cable and select the proper wall thickness from Table I.

NOTE.—It is preferable, both in Paragraphs (*a*) and (*b*) above, to select a cable with a higher voltage rating than obtained using the above procedure, both as a measure of safety and to allow the substitution of a larger transformer without necessitating the replacement of the cable.

71

The thickness of insulation for the various systems shall be determined as follows:

A2. (*a*) For three-phase systems with grounded or ungrounded neutral, use thickness values in accordance with Table I.

(*b*) For single or two-phase systems up to and including 5000 v., use thickness values in accordance with Table I, as specified for grounded neutral. Where it is not definitely specified that a line operates as an isolated single or two-phase system it shall be considered as a branch of a grounded three-phase circuit and the thickness of insulation shall be that required by the phase-to-phase voltage of this grounded, three-phase circuit as given in Table I.

(*c*) For single or two-phase systems operating at over 5000 v. with one side grounded, multiply the circuit voltage (phase-to-phase) by 1.73 and use the resulting voltage value to select the corresponding insulation thickness for grounded neutral as specified in Table I.

(*d*) For single or two-phase systems operating at over 5000 v. with the center grounded, multiply the circuit voltage (phase-to-phase) by 0.866 $(\frac{1}{2}\sqrt{3})$ and use the resulting voltage value to select the corresponding insulation thickness for grounded neutral as specified in Table I.

(*e*) For single or two-phase ungrounded systems operating at over 5000 v., multiply the circuit voltage (phase-to-phase) by 0.866 $(\frac{1}{2}\sqrt{3})$ and use the resulting voltage value to select the corresponding insulation thickness for ungrounded neutral as specified in Table I.

(*f*) For direct-current systems, consider them the same as single-phase alternating-current systems in accordance with Paragraph A2 (*b*) above.

TENTATIVE SPECIFICATIONS

FOR

INSULATED WIRE AND CABLE: PERFORMANCE RUBBER COMPOUND[1]

A.S.T.M. Designation: D 353 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1932; REVISED, 1933, 1934, 1935, 1936, 1937.[2]

Scope

1. (*a*) These specifications cover wire and cable insulated with a vulcanized rubber compound. The suitability of the compound as insulation shall be determined solely by physical and electrical tests. The compound shall be called performance rubber compound.

(*b*) Except for the rubber insulation and methods of testing, wire and cable supplied under these specifications shall conform to the Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (A.S.T.M. Designation: D 27 - 37 T) of the American Society for Testing Materials.[3]

INSULATION

Workmanship

2. (*a*) The insulation shall be homogeneous in character, tough, elastic and applied concentrically about the conductor and shall fit tightly thereto. Where the insulation is applied in more than one layer adjacent layers shall be vulcanized into a homogeneous mass.

(*b*) *Shielding.*—Braided cables not supported on insulators when operating at 6001 v. and over with the neutral grounded or 3501 v. and over with the neutral ungrounded shall be shielded or suitably protected against corona. For special installations, such as braided cables connected to an overhead line, shielding may be provided at voltages lower than those given herein.

All lead-covered multiple conductor cables operating at 6001 v. and over with the neutral ungrounded shall be shielded or suitably protected against corona.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

[2] Latest revision accepted by Committee E-10 on Standards, August 26, 1937.

[3] See p. 1070.

The shielding shall consist of a tinned copper tape at least 3 mils thick or braid of fine tinned copper wires.

(*c*) The insulation of braided wires up to No. 7 A. w. g., inclusive, shall be covered with a single cotton braid. On larger sizes the fibrous covering shall consist of a cable tape and cotton braid or two cotton braids. The cable tape may be applied before vulcanization. In multiple conductor cables the individual conductors shall not be braid-covered unless specified.

(*d*) Where repairs or joints are made in the insulation, the work shall be done in such manner that the repaired part of the joint, and all parts affected in the process, shall be as strong and durable electrically and mechanically as the remainder of the insulation and shall not exceed the limitations on the thickness specified in Section 5.

MANUFACTURE

Composition

3. The insulation shall consist of a properly vulcanized rubber compound that will meet the tests hereinafter specified.

PHYSICAL PROPERTIES

Physical Properties

4. The insulation shall conform to the following requirements as to physical properties:

	MAXIMUM	MINIMUM
Tensile strength, lb. per sq. in.	..	1200
Tensile stress at 200 per cent elongation, lb. per sq. in.	..	300
Elongation at rupture, per cent	..	400
Set in 2-in. gage length, in.	$\frac{3}{8}$	....
Depreciation in tensile strength and elongation after oxygen bomb test, per cent	25	....
Depreciation in tensile strength and elongation after Geer oven test, per cent	15	....

THICKNESS

Thickness

5. (*a*) The average thickness of the insulation shall be not less than that given in Table I. The minimum thickness shall be not less than 95 per cent of the thickness required by Table I for insulations less than $\frac{4}{64}$ in. in thickness and not less than 90 per cent of the specified thickness for insulations of $\frac{4}{64}$ in. and greater.

(*b*) The thicknesses in Table I apply to single conductor cable and the individual conductors of multiple conductor cables, leaded or braided, except special applications such as aerial, non-leaded submarine, and vertical riser cable.

NOTE.—In Appendix I will be found an interpretation of the insulation thicknesses given in Table I.

TABLE I.—THICKNESS OF INSULATION AND ALTERNATING TEST VOLTAGE.

NOTE 1.—Rated circuit voltage of alternating-current circuits is the phase-to-phase voltage.

NOTE 2.—For an intermediate size of wire or an intermediate rated circuit voltage, the thickness and test voltage shall be that for the next larger size or higher rated circuit voltage.

NOTE 3.—For non-leaded submarine cables, $\frac{1}{32}$ in. should be added to the specified respective walls of insulation in this table for all sizes and voltages.

NOTE 4.—Rated circuit voltages from 0 to 5000 v. in this table apply to wire for use on grounded and ungrounded circuits.

NOTE 5.—Above 5000 v. alternating-current, special high-voltage rubber compounds are preferred according to manufacturer's recommendations.

Rated Circuit Voltage	Size of Conductor, A. w. g. Numbers or Circular Mils	Insulation Thickness on each Conductor, sixty-fourths of an inch	Test Voltage
0 to 300	No. 18 to No. 16	2	1 000
0 to 600	No. 14 to No. 9	3	3 000
	No. 8 to No. 2	4	3 500
	No. 1 to No. 0000	5	4 000
	225 000 to 500 000 cir. mils	6	5 000
	525 000 to 1 000 000 cir. mils	7	6 000
	Over 1 000 000 cir. mils	8	7 000
601 to 1000	No. 14 to No. 8	4	5 000
	No. 7 to No. 2	5	6 000
	No. 1 to No. 0000	6	7 500
	225 000 to 500 000 cir. mils	7	9 000
	525 000 to 1 000 000 cir. mils	8	10 000
	Over 1 000 000 cir. mils	9	11 000
1001 to 2000	No. 14 to No. 8	5	6 000
	No. 7 to No. 2	6	7 500
	No. 1 to No. 0000	7	9 000
	225 000 to 500 000 cir. mils	8	10 000
	525 000 to 1 000 000 cir. mils	9	11 000
	Over 1 000 000 cir. mils	9	11 000
2001 to 3000	No. 14 to No. 8	7	9 000
	No. 7 to No. 2	8	10 000
	No. 1 to No. 0000	8	10 000
	225 000 to 500 000 cir. mils	9	11 000
	525 000 to 1 000 000 cir. mils	9	11 000
	Over 1 000 000 cir. mils	10	12 500
3001 to 4000	No. 14 to No. 8	9	11 000
	No. 7 to No. 2	9	11 000
	No. 1 to No. 0000	9	11 000
	225 000 to 500 000 cir. mils	10	12 500
	525 000 to 1 000 000 cir. mils	10	12 500
	Over 1 000 000 cir. mils	11	13 500
4001 to 5000	No. 14 to No. 8	10	12 500
	No. 7 to No. 2	10	12 500
	No. 1 to No. 0000	10	12 500
	225 000 to 500 000 cir. mils	11	13 500
	525 000 to 1 000 000 cir. mils	11	13 500
	Over 1 000 000 cir. mils	12	15 000
5001 to 6000 (grounded)	No. 14 to No. 0000	10	12 500
	225 000 to 1 000 000 cir. mils	11	13 500
	Over 1 000 000 cir. mils	12	15 000
5001 to 6000 (ungrounded)	No. 14 to No. 0000	12	15 000
	225 000 to 1 000 000 cir. mils	12	15 000
	Over 1 000 000 cir. mils	13	16 500
6001 to 7000 (grounded)	No. 8 to No. 0000	11	13 500
	225 000 to 1 000 000 cir. mils	11	13 500
	Over 1 000 000 cir. mils	12	15 000
6001 to 7000 (ungrounded)	No. 8 to No. 0000	14	17 500
	225 000 to 1 000 000 cir. mils	14	17 500
	Over 1 000 000 cir. mils	15	19 000
7001 to 8000 (grounded)	No. 8 to No. 0000	12	15 000
	225 000 to 1 000 000 cir. mils	12	15 000
	Over 1 000 000 cir. mils	13	16 500
7001 to 8000 (ungrounded)	No. 8 to No. 0000	16	20 000
	225 000 to 1 000 000 cir. mils	16	20 000
	Over 1 000 000 cir. mils	17	21 000

ELECTRICAL PROPERTIES

High Voltage Test

6. Each coil, reel, or length of wire or cable after vulcanization shall successfully withstand the application of an alternating voltage of not less than that prescribed in Table I for a period of 5 minutes. (See Sections 30 and 31.)

Insulation Resistance

7. Each coil, reel, or length of wire or cable after being subjected to the high voltage test specified in Section 6 shall have an insulation resistance not less than that given in Table II.

Moisture Absorption.

8. (*a*) The increase in the specific inductive capacity of the insulation shall be used as an indication of the amount of moisture absorbed.

(*b*) The specific inductive capacity after 24-hr. immersion in water shall not exceed six. The increase between the ends of the first and the fourteenth days shall not exceed 20 per cent and that between the ends of the seventh and fourteenth days, 5 per cent.

(*c*) The specific inductive capacity shall be determined at either 1000 or 60 cycles. Where measured at 1000 cycles, the voltage impressed upon the conductor shall not exceed 10 volts. Where measured at 60 cycles, the voltage impressed upon the conductor shall be equivalent to a stress of between 30 to 40 volts per mil of insulation.

(*d*) The measurements shall be made as described in Section 35.

METHODS OF TESTING

TENSILE STRENGTH, SET, AND ELONGATION AT RUPTURE

Sampling for Physical Tests

9. (*a*) Wire up to and including 250,000 cir. mils in size shall be sampled in accordance with the following requirements:

	NUMBER OF SAMPLES
2000 ft. and under	none
2001 to 10,000 ft.	1 for each 2000 ft. or fraction thereof
10,001 to 100,000 ft.	5 plus 1 for each 10,000 ft. additional over 10,000 ft.
100,001 to 500,000 ft.	14 plus 1 for each 25,000 ft. over 100,000 ft.

(*b*) For wire larger than 250,000 cir. mils, one sample shall be selected from quantities between 1000 and 2000 ft. and for larger quantities double those specified in Paragraph (*a*).

Test Specimens

10. (*a*) *Size.*—The test specimen may be the entire section of the insulation in the case of small wires, but in the case of wire and cable

of No. 6 A. w. g. and larger or with an insulation thickness greater than $\frac{3}{32}$ in., samples approximately square in section shall be cut from the insulation with a cross-section not greater than 0.025 sq. in. The test specimen shall be as free as possible from surface incisions and imperfections.

TABLE II.—INSULATION RESISTANCE, MEGOHMS-1000 FT. AT 60° F. (15.5° C.).

NOTE 1.—This table is based on a constant, $K = 21\,120$ in the following formula:

$$R = K \log_{10}\frac{D}{d};$$ where R = resistance in megohms-1000 ft., K = constant, D = diameter over insulation, on each conductor, and d = diameter over conductor.

NOTE 2.—For an intermediate size, the insulation resistance shall be that of the next larger size.

Size of Conductor, A. w. g. Numbers or Circular Mils	Solid or Stranded	Diameter in Inches	Insulation Thickness in Sixty-fourths of an Inch									
			2	3	4	5	6	7	8	9	10	12
No. 18	Solid	0.040	8 600	11 100	....	....	....	....	....	....	....	....
No. 16	Solid	0.051	7 300	9 500	....	....	....	....	....	....	....	....
No. 14	Solid	0.064	....	8 300	9 900	11 300	12 550	13 650	14 600	15 450	16 250	17 650
No. 12	Solid	0.081	....	7 050	8 550	9 850	11 000	12 000	12 900	13 750	14 500	15 850
No. 10	Solid	0.102	....	6 000	7 350	8 500	9 600	10 500	11 350	12 100	12 850	14 150
No. 8	Solid	0.128	....	....	6 200	7 300	8 300	9 150	9 950	10 650	11 350	12 550
No. 6	Stranded	0.184	....	....	4 750	5 600	6 400	7 200	7 900	8 500	9 100	10 200
No. 4	Stranded	0.232	....	....	3 900	4 700	5 450	6 100	6 700	7 250	7 800	8 850
No. 2	Stranded	0.292	....	....	3 250	3 900	4 550	5 150	5 650	6 150	6 650	7 600
No. 1	Stranded	0.332	....	....	....	3 550	4 100	4 650	5 150	5 600	6 100	6 950
No. 0	Stranded	0.373	....	....	....	3 200	3 750	4 200	4 700	5 150	5 550	6 350
No. 00	Stranded	0.418	....	....	....	2 900	3 400	3 850	4 300	4 700	5 100	5 850
No. 000	Stranded	0.470	....	....	....	2 600	3 100	3 500	3 900	4 250	4 650	5 400
No. 0000	Stranded	0.528	....	....	....	2 400	2 800	3 150	3 550	3 900	4 200	4 900
250 000 cir. mils	Stranded	0.575	....	....	....	....	2 600	2 950	3 300	3 650	4 000	4 600
350 000 cir. mils	Stranded	0.681	....	....	....	....	2 250	2 550	2 850	3 150	3 450	4 000
500 000 cir. mils	Stranded	0.814	....	....	....	....	1 900	2 200	2 450	2 700	2 950	3 450
750 000 cir. mils	Stranded	0.998	....	....	....	....	....	1 800	2 050	2 250	2 500	2 900
1 000 000 cir. mils	Stranded	1.152	....	....	....	....	....	1 600	1 800	2 000	2 200	2 600
1 250 000 cir. mils	Stranded	1.289	....	....	....	....	....	....	1 600	1 800	2 000	2 350
1 500 000 cir. mils	Stranded	1.412	....	....	....	....	....	....	1 500	1 650	1 850	2 150
1 750 000 cir. mils	Stranded	1.526	....	....	....	....	....	....	1 400	1 550	1 700	2 000
2 000 000 cir. mils	Stranded	1.631	....	....	....	....	....	....	1 300	1 500	1 600	1 900

Size of Conductor, A. w. g. Numbers or Circular Mils	Solid or Stranded	Diameter in Inches	Insulation Thickness in Sixty-fourths of an Inch				
			13	14	15	16	17
No. 8	Solid	0.128	13 100	13 600	14 100	14 600	14 900
No. 6	Stranded	0.184	10 650	11 150	11 500	12 000	12 450
No. 4	Stranded	0.232	9 300	9 700	10 100	10 500	10 900
No. 2	Stranded	0.292	7 950	8 400	8 800	9 150	9 500
No. 1	Stranded	0.332	7 350	7 650	8 000	8 400	8 750
No. 0	Stranded	0.373	6 750	7 050	7 400	7 800	8 100
No. 00	Stranded	0.418	6 200	6 500	6 900	7 150	7 500
No. 000	Stranded	0.470	5 700	6 150	6 350	6 650	6 900
No. 0000	Stranded	0.528	5 200	5 500	5 800	6 050	6 350
250 000 cir. mils	Stranded	0.575	4 900	5 200	5 450	5 750	5 950
350 000 cir. mils	Stranded	0.681	4 250	4 500	4 700	4 950	5 200
500 000 cir. mils	Stranded	0.814	3 700	3 950	4 150	4 350	4 550
750 000 cir. mils	Stranded	0.998	3 100	3 300	3 500	3 700	3 900
1 000 000 cir. mils	Stranded	1.152	2 750	2 950	3 100	3 300	3 450
1 250 000 cir. mils	Stranded	1.289	2 500	2 650	2 850	3 000	3 150
1 500 000 cir. mils	Stranded	1.412	2 300	2 450	2 600	2 700	2 950
1 750 000 cir. mils	Stranded	1.526	2 150	2 300	2 450	2 600	2 750
2 000 000 cir. mils	Stranded	1.631	2 050	2 200	2 300	2 450	2 600

NOTE.—The removal of the rubber insulation can be greatly accelerated and in most cases a test specimen which is an entire cross-section can be obtained free from surface incisions and imperfections by means of metallic mercury. The

mercury should be introduced at one end of the sample between the insulation and the tinned surface of the conductor, and the sample inclined on a support with the end to which the mercury is applied at the top. The separation of the rubber insulation results from the amalgamation of the tin of the conductor with the mercury. The amalgamation is assisted by first immersing and rubbing the tinning on the exposed end of the conductor in the mercury.

(*b*) *Condition and Age.*—Specimens shall not be heated, immersed in water or subjected to any mechanical or chemical treatment not specifically prescribed in these specifications.

Specimens for accelerated aging tests shall be taken from the completed wire after the application of the specified covering and shall be aged with all such covering removed.

No tests shall be made within 24 hours nor later than 60 days after vulcanization unless agreed to by the manufacturer.

(*c*) *Buffing.*—In the event of any irregularities on the surface of the test specimen including corrugations from large strands, it shall be made smooth and of uniform thickness by buffing.

Calculation of Area

11. Calculation of the area of the test specimens shall be made as follows:

(*a*) Where the total cross-section of the insulation is used, the area shall be taken as the difference between the area of the circle whose diameter is the average outside diameter of the insulation and the area of the conductor. The area of the stranded conductor shall be calculated from its maximum diameter.

(*b*) Where a segment or sector of the insulation has to be taken in the case of a large wire or cable or where the insulation is thin, the area shall be calculated as the thickness times the width. (This applies either to a straight test specimen or one stamped out with a die, and assumes that corrugations have been removed by buffing.)

Temperature at Test

12. Physical tests shall be made at a room temperature not less than 65° F. (18.3° C.) nor more than 90° F. (32.2° C.) and the test specimen shall have been kept at the room temperature not less than 30 minutes prior to the test.

Tension Testing Machine

13. The tension testing machine shall be power-driven and preferably of the pendulum type. The machine shall be accurate within 1 per cent of the breaking load. A spring balance type of apparatus may be used if provided with a device which will indicate the actual maximum load at which rupture takes place and if provision is made to prevent recoil of the spring.

Tensile Strength Tests

14. The tensile strength test shall be made on a specimen which has not been previously stretched. The specimen shall have a length of not less than 6 in., and shall be marked with gage marks 2 in. apart and placed in the jaws of the testing machine with a maximum distance between jaws of 4 in. The specimen shall be stretched at the rate of 20 in. per minute (jaw speed) until it breaks. The test specimen shall break between the gage marks. The tensile strength shall be calculated upon the area of the original specimen.

Tensile Stress Test

15. The tensile stress test shall be made simultaneously with the tensile strength test by recording the load when the 2-in. gage length marks are 6 in. apart, that is, at 200 per cent elongation. The tensile stress shall be calculated upon the area of the original specimen.

Elongation at Rupture

16. Elongation at rupture shall be determined simultaneously with the tensile strength test by reading the distance in inches between gage marks at the breaking point. The elongation at rupture shall be taken as the distance between gage marks at rupture less 2 in. (the original gage length). The percentage of elongation at rupture is the elongation in inches divided by the original gage length (2 in.) and multiplied by 100 to express as a percentage.

Set Test

17. The set test shall be made on a separate test specimen having a length of not less than 6 in. and marked with gage marks 2 in. apart. The specimen shall be placed in the jaws of the testing machine with a maximum distance between jaws of 4 in. and shall be stretched at the rate of 20 in. per minute (jaw speed) until the gage marks are 6 in. apart. The test specimen shall then be released within 5 seconds and the distance between gage marks shall be determined 1 minute after the beginning of release. The set is the difference between this length and the original 2-in. gage length.

Retests, Physical Properties

18. Where tested in accordance with Sections 14, 15, 16 and 17, if a specimen fails to conform to any one of the requirements for tensile strength, tensile stress, set, or elongation at rupture specified in Section 4, two additional specimens shall be taken from the same sample and tested, and the average of the results shall determine whether the material conforms to the requirements.

Aging Tests

Sampling for Aging Tests

19. For aging tests the wire shall be sampled as follows:

No samples shall be selected where the quantity is less than 2000 ft. of wire or cable.

For sizes less than 250,000 cir. mils one sample shall be selected for each quantity ordered between 2000 ft. and 50,000 ft. of wire or cable and one additional sample for each additional 50,000 ft. thereafter.

For sizes 250,000 cir. mils and over one sample shall be selected for each quantity ordered between 2000 ft. and 25,000 ft. of wire or cable and one additional sample for each additional 25,000 ft. thereafter.

Test Specimens

20. Nine test specimens of similar size and shape shall be prepared from each sample selected in accordance with Section 19, three for the determination of the initial or unaged properties, three for the oxygen bomb test and three for the Geer oven test. One specimen of each three shall be tested and the other two held as spares except that where only one sample is selected all three specimens shall be tested and the average of the results reported.

Aging Test Conditions

21. (*a*) In the case of wire and cable smaller than No. 6 A.w.g. with an insulation thickness less than $\frac{3}{32}$ in., the insulation shall be heated in the oxygen bomb and in the Geer oven without removing the conductor.

(*b*) In the case of wire and cable of No. 6 A.w.g. and larger or with an insulation thickness greater than $\frac{3}{32}$ in., samples approximately square in section shall be cut from the insulation with a cross-section not greater than 0.025 sq. in.

(*c*) Die-cut samples shall be buffed before being subjected to the accelerated aging test wherever the thickness of the sample is $\frac{3}{32}$ in. or greater after buffing.

(*d*) The test specimens shall have no protective covering and shall be suspended vertically in such a manner that they will not come in contact with each other or with the sides of the bomb or oven.

(*e*) Physical tests on both the aged and unaged specimens shall be made at the same time. The aged specimens shall have a rest period of not less than 16 hours nor more than 48 hours between the completion of the aging test and determination of physical properties.

(*f*) Should the first set of specimens selected under Paragraph (*a*) fail to meet the specifications, two other sets of specimens shall be tested and if either of these sets fails, the coils, reels or lengths will be rejected.

Oxygen Bomb Test

22. The test specimens shall be heated in an atmosphere of oxygen at a pressure of 300 lb. per sq. in. at a temperature of from 157 to 159° F. for a period of 96 hours. The weight of the rubber in the bomb shall not be over 2 g. per cu. in. of bomb space. The bomb pressure shall be reduced at a uniform rate, requiring at least two minutes, when specimens are removed. The bomb temperature shall be recorded automatically on a chart.

Geer Oven Test

23. The test specimens shall be heated at a temperature of from 157 to 159° F. for a period of 96 hours in an oven having a free circulation of fresh air. The oven temperature shall be recorded automatically on a chart.

Rejection

24. Where ten or more samples are selected in any inspection lot, all coils, reels or lengths shall be rejected if more than 10 per cent of the samples fail. If 10 per cent or less fail, each coil, reel or length may be tested and shall be accepted or rejected upon the results of such individual tests. Where the number of samples selected in any inspection lot is less than ten, all coils, reels or lengths shall be rejected if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel or length may be tested and shall be accepted or rejected upon the results of such individual tests.

THICKNESS MEASUREMENTS

Apparatus

25. The thickness measurements may be made with any type of micrometer reading to 0.001 in., suitable for measurements of this character.

Number of Measurements

26. Where the lot of wire to be inspected consists of two coils or reels or less, at least one determination of the thickness shall be made on each coil or reel. Where the lot consists of more than two coils or reels and less than 20 coils or reels, at least one determination of the thickness shall be made on each of two coils or reels taken at random. If the lot consists of 20 or more coils or reels, not less than

10 per cent of the coils or reels shall be selected at random and at least one determination of the thickness made on each coil or reel so selected.

Procedure

27. The average thickness of the insulation shall be taken as one-half the difference between the mean of the maximum and minimum diameters measured at any point and the average diameter of the conductor measured at the same point. The minimum thickness shall be taken as the difference between a measurement made over the conductor plus the thinnest wall and the diameter of the conductor. (The first measurement is made by "slicing off" the heavier side of the insulation.)

In the case of multiple-conductor cable, the measurements shall be made on the individual wires before being cabled.

Rejection

28. If the thickness of the insulation of any coil or reel is found to be less than the specified value, that coil or reel shall be rejected and a thickness measurement on each of the remaining coils or reels shall be made.

ELECTRICAL TESTS

Place

29. Electrical tests of wire and cable shall be made at the place of manufacture.

High Voltage Test

30. The high voltage test shall be made with alternating potential from a transformer and generator of ample capacity and in no case less than 5 kva. The frequency of the test voltage shall be not greater than 100 cycles and shall have a wave shape approximating as closely as possible a sine curve

Rate of Application

31. The initially applied voltage shall be not greater than the rated voltage and the rate of increase shall be approximately uniform and not over 100 per cent in 10 seconds nor less than 100 per cent in 60 seconds.

High Voltage Testing

32. (*a*) *General.*—Where insulated conductors are subjected to a high voltage test in water they shall be immersed in a grounded water bath and tested while still immersed.

(*b*) *Single Conductor.*—Single conductors shall be tested after 12 hr. immersion and without covering except a tape or braid applied before vulcanization.

Rubber sheathed conductors shall be immersed and tested after the sheath is applied.

Where a metallic sheath is applied, the conductors shall be retested against the sheath.

Single-conductor cables shall be tested between conductor and sheath or water in which they are immersed at the test voltage required by Table I.

Table III.—Temperature Coefficients.

Temperature		Temperature Coefficient	Temperature		Temperature Coefficient
Deg. Fahrenheit	Deg. Centigrade		Deg. Fahrenheit	Deg. Centigrade	
46	7.8	0.69	61	16.1	1.03
47	8.3	0.71	62	16.7	1.05
48	8.9	0.73	63	17.2	1.08
49	9.4	0.75	64	17.8	1.11
50	10.0	0.77	65	18.3	1.14
51	10.6	0.79	66	18.9	1.17
52	11.1	0.81	67	19.4	1.20
53	11.7	0.83	68	20.0	1.23
54	12.2	0.85	69	20.6	1.26
55	12.8	0.88	70	21.1	1.30
56	13.3	0.90	71	21.7	1.33
57	13.9	0.92	72	22.2	1.37
58	14.4	0.94	73	22.8	1.40
59	15.0	0.97	74	23.3	1.44
60	15.6	1.00	75	23.9	1.48

(*c*) *Multiple Conductor.*—The individual conductors of multiple conductor cables shall be tested before assembling as prescribed in Paragraph (*b*).

After assembly each conductor shall be tested against the adjacent conductors. Where a metallic sheath is applied each conductor shall in addition be tested against the sheath.

Cables having a rubber or other non-metallic waterproof sheath shall be immersed for 12 hr. and the conductors tested against ground. Cables covered with cable tape and braid or double braid with a weather-resistant finish shall not be immersed for testing.

Multiple-conductor cables after assembly shall be tested at the test voltage required by Table I.

Insulation Resistance

33. (*a*) The insulation resistance shall be measured after the high voltage test has been made and while the wire or cable is still immersed in water. The measurement shall be made after one

minute electrification with a continuous e.m.f. of from 100 to 500 volts, the conductor being maintained negative to the water. The temperature of the water shall be within the limits given in Table III.

Temperature Coefficient

(*b*) If the temperature at which the insulation resistance was measured differs from 60° F. (15.6° C.), the resistance shall be reduced to that at 60° F. (15.6° C.) by multiplying the measured value by the coefficient in Table III corresponding to the temperature at which the measurement was made.

Moisture Absorption Test

34. (*a*) A 15-ft. sample of the insulated wire or cable shall be taken after vulcanization and prior to the application of any coverings except tape applied before vulcanizing. Such cable tape shall be removed before making the moisture absorption test. The middle 10 ft. of the sample shall be immersed in distilled water for a period of 14 days with the $2\frac{1}{2}$-ft. portion at each end kept above the water as leakage insulation. The three measurements specified in Section 8 shall be taken with the water at the same temperature. The water shall be maintained at room temperature, but not less than 70° F. (21° C.).

(*b*) The specific inductive capacity of the insulation shall be determined at commercial frequencies and shall be calculated as follows:

$$\text{Specific inductive capacity} = 13\,600\; C \log_{10} \frac{D}{d}$$

where C = the capacity in microfarads of 10 ft. of sample;
D = the diameter over insulation; and
d = the diameter over conductor.

The moisture absorption test shall be made only when requested by the purchaser.

Rejection

35. Each coil, reel or length which fails to comply with the electrical requirements of these specifications shall be rejected.

APPENDIX

INTERPRETATION OF INSULATION THICKNESSES OF TABLE I

For series-street-lighting cable, use the following procedure:

A1. (*a*) If the transformer is not equipped with protectors, use the open circuit secondary voltage of the transformer as rated voltage of the cable and select the proper wall thickness from Table I.

(*b*) If the transformer is equipped with protectors, use the closed or loaded secondary voltage of the transformer as the rated circuit voltage of the cable and select the proper wall thickness from Table I.

NOTE.—It is preferable, both in Paragraphs (*a*) and (*b*) above, to select a cable with a higher voltage rating than obtained using the above procedure, both as a measure of safety and to allow the substitution of a larger transformer without necessitating the replacement of the cable.

The thickness of insulation for the various systems shall be determined as follows:

A2. (*a*) For three-phase systems with grounded or ungrounded neutral, use thickness values in accordance with Table I.

(*b*) For single or two-phase systems up to and including 5000 v., use thickness values in accordance with Table I, as specified for grounded neutral. Where it is not definitely specified that a line operates as an isolated single or two-phase system it shall be considered as a branch of a grounded three-phase circuit and the thickness of insulation shall be that required by the phase-to-phase voltage of this grounded, three-phase circuit as given in Table I.

(*c*) For single or two-phase systems operating at over 5000 v. with one side grounded, multiply the circuit voltage (phase-to-phase) by 1.73 and use the resulting voltage value to select the corresponding insulation thickness for grounded neutral as specified in Table I.

(*d*) For single or two-phase systems operating at over 5000 v. with the center grounded, multiply the circuit voltage (phase-to-phase) by 0.866 ($\frac{1}{2}\sqrt{3}$) and use the resulting voltage value to select the corresponding insulation thickness for grounded neutral as specified in Table I.

(*e*) For single or two-phase ungrounded systems operating at over 5000 v., multiply the circuit voltage (phase-to-phase) by 0.866 ($\frac{1}{2}\sqrt{3}$) and use the resulting voltage value to select the corresponding insulation thickness for ungrounded neutral as specified in Table I.

(*f*) For direct-current systems, consider them the same as single-phase alternating-current systems in accordance with Paragraph A2 (*b*) above.

TENTATIVE METHOD FOR AIR PRESSURE HEAT TEST OF VULCANIZED RUBBER[1]

A.S.T.M. Designation: D 454 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. (*a*) This method is intended for use as an accelerated test applicable for estimating the probable life of rubber compounds designed for high-temperature service. It is not suitable for rubber products which have not been specifically designed to withstand elevated temperatures in service. No direct correlation between the test and actual life in service should be implied but when the test is used comparatively between rubber compounds of similar type, the measured changes give valuable indication as to the relative ability of each compound to withstand service conditions involving the same factors as those employed in the test.

(*b*) Except as may be otherwise specified in this method, the requirements of the Tentative Methods of Physical Testing of Rubber Products (General Requirements) (A.S.T.M. Designation: D 15 - 35 T) of the American Society for Testing Materials[2] shall be complied with and are made a part of this method.

(*c*) In case of conflict between the provisions of this method and those of detailed specifications or methods of test for a particular material, the latter shall take precedence.

Type of Test

2. (*a*) The air pressure heat test for rubber consists of exposing test specimens having previously determined physical properties to the deteriorating influence of air at specified elevated temperature and pressure for known periods, after which the physical properties are again measured and the changes noted.

(*b*) The physical properties used to measure the deterioration of the rubber, in addition to visual and manual inspection, are tensile stress at a specified elongation or tensile strength and ultimate elongation, or both. Except as otherwise specified in this method, the determination of these properties before and after each exposure period shall be carried out in accordance with the Tentative Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412 – 36 T) of the American Society for Testing Materials.[1]

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1153 (1935); also 1937 Book of A.S.T.M. Tentative Standards, p. 1260.

Apparatus

3. The air-pressure chamber shall consist of a metal vessel designed to maintain an internal atmosphere of air under known pressure with provisions for placing rubber specimens within it and subjecting them to controlled uniform temperature. The equipment shall conform to the following requirements:

(*a*) The size and shape of the chamber shall be optional but shall be such that the specimens may be suspended therein vertically without undue crowding and without touching each other or the sides of the chamber.

(*b*) The operating temperature shall be 126.7 C. ± 1 C. (260 F. ± 1.8 F.) determined by measurement at the center of the suspended specimens and shall be automatically controlled by means of thermostatic regulation.

(*c*) The source and means of heating shall be optional but the uniformity of temperature distribution at various points within the pressure chamber shall be verified by actual check since the test is sensitive to small temperature variations. Local overheating or "dead spots" shall be avoided. Care shall also be taken in the design of the equipment to avoid direct radiation of heat on the samples.

(*d*) The apparatus and method of heating shall be so designed that the interval required for the chamber to reach the operating temperature at the beginning of a test shall be as short as possible. By proper precautions this lag may be reduced to less than 5 per cent of the usual minimum exposure periods. Provision shall also be made for rapid closing and opening of the apparatus for introduction or removal of specimens.

(*e*) The air pressure shall be maintained at 80 lb. per sq. in. ± 2.0 lb. per sq. in. during the exposure periods. Automatic regulation is recommended.

(*f*) Suitable provision shall be made by separation, filtration, or otherwise for removal of oil, dirt, and moisture from the air entering the pressure chamber. Care shall also be taken to avoid any other introduction of oil or grease into the pressure chamber.

(*g*) No copper or brass parts shall be exposed to the atmosphere used in the pressure chamber.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1105 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1266.

(*h*) The pressure chamber shall be equipped with a reliable safety valve set for release at a pressure of not more than 200 lb. per sq. in.

NOTE: *Caution.*—Adequate safety provisions are important when heating oxidizable organic materials in air under pressure since the rate of oxidation may become very rapid in some cases, particularly if large surface area is exposed.

Test Specimens

4. (*a*) Dumbbell-shaped test specimens, prepared as described in the Tentative Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412 – 36 T) of the American Society for Testing Materials,[1] shall be considered standard. Their form shall be such that no mechanical, chemical, or heat treatment will be required after exposure in the pressure chamber. If buffing is necessary, it shall be performed prior to exposure.

(*b*) The dimensions of the test specimens for calculating the physical properties shall be measured prior to exposure in the pressure chamber. Care shall be taken that the material used for marking the gage lines on the specimens is not detrimental to the rubber during the heat treatment, causing the specimens to break at the marks prematurely during subsequent test. Only specimens of similar dimensions having approximately the same exposed areas may be compared with each other.

(*c*) At least three test specimens shall be used to determine the average original physical properties of each sample and also three or more specimens of the same material for each exposure period of the test.

Tests of Original Specimens

5. The tensile stress at a specified elongation or tensile strength and ultimate elongation, or both, of the original unexposed specimens shall be determined within 48 hr. of the start of the heat treatment. Results on specimens which break outside of the straight reduced section, or which are found to be imperfect, shall be discarded and retests shall be made.

Air Pressure Heat Treatment

6. (*a*) The specimens for exposure shall be suspended vertically in the pressure chamber after it has been preheated to the operating temperature. Simultaneous exposure of a mixed group of different compounds shall be avoided, if possible. For instance, high-sulfur compounds should not be exposed with low-sulfur compounds nor those containing antioxidants with those which do not, since some migration is known to occur under the conditions of the test.

(*b*) The exposure period shall be considered to start when the specimens are placed in the heated chamber which shall be closed immediately and the air pressure applied. This entire operation shall not require longer than 3 min. However, a record shall be kept of the time interval elapsing from the starting time until the temperature of the chamber reaches 126.7 C.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1105 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1266.

(260 F.). If this interval exceeds 5 per cent of the total exposure time, approximate correction shall be made by adding one-half of the interval to the exposure period. The exposure shall be continuous for the specified time without pressure reduction or opening of the chamber for introduction or removal of specimens.

(*c*) The selection of suitable periods of exposure will depend on the rate of deterioration of the particular material being tested and should be such that the deterioration will not be so great as to prevent determination of the final physical properties. Intervals frequently used are 3, 5, 8, 12, 20 and 30 hr. In experimental work, it is desirable to employ a range of periods so as to determine the rate of deterioration but, for routine tests of known materials and for purchase acceptance purposes, fewer intervals or even a single period may suffice.

(*d*) At the termination of the exposure period, the air pressure shall be released gradually, the operation requiring at least 2 min. so as to avoid possible formation of porosity in the specimens which shall then be immediately removed from the pressure chamber. The specimens shall be cooled to room temperature on a flat surface and allowed to rest not less than 16 hr. nor more than 48 hr. before determination of the physical properties.

Tests of Exposed Specimens

7. The tensile stress at a specified elongation or tensile strength and ultimate elongation, or both, of the specimens exposed for different periods shall be determined as the intervals terminate, except that it shall be permissible to accumulate specimens for testing together when this does not conflict with the specified rest period. In determining the final physical properties, the values shall be the average of results from at least two specimens, including that one giving the highest value for ultimate tensile strength and any which check that value within 10 per cent. Results of tests of all other specimens shall be discarded. In the event that discarding of exposed specimens both from faulty breaks and failure to check does not leave two satisfactory determinations, additional specimens shall be exposed and tested until two or more check results from properly broken specimens are obtained. After completion of the tests, the broken specimens shall be examined visually and manually and their condition noted.

Calculation of Results

8. The results of the air pressure heat test for each exposure period shall be expressed in the form of percentage of deterioration in each physical property (tensile strength, ultimate elongation or tensile stress), calculated as follows:

$$\text{Percentage of deterioration} = \frac{(\text{Original value} - \text{Value after exposure})}{\text{Original value}} \times 100$$

Report

9. The report shall include the following:

   (*a*) The results calculated in accordance with Section 8,
   (*b*) All observed and recorded data on which the calculations are based,
   (*c*) Description of the apparatus,
   (*d*) The exposure period,
   (*e*) Statement of condition of exposed specimens,
   (*f*) Dimensions of test specimens,
   (*g*) The duration, temperature and date of vulcanization of the rubber, if known, and
   (*h*) Dates of original and final determination of physical properties.

TENTATIVE METHODS OF TEST
FOR
COMPRESSION SET OF VULCANIZED RUBBER[1]

A.S.T.M. Designation: D 395 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1935; REVISED, 1936, 1937.

Scope

1. These methods are designed for testing vulcanized rubber parts which are to be assembled in such a manner that the rubber will be subjected to compressive stresses or shear. They are applicable particularly to the rubber used in machinery mountings and vibration dampers.

Compression Set

2. For the purposes of these tests, compression set of rubber shall be considered as the residual decrease in thickness, expressed as the percentage of the original thickness, of a test specimen measured 30 min. after removal from a suitable loading device in which the specimen had been subjected for a definite time to compressive deformation under specified conditions of load application and temperature.

Types of Tests

3. (*a*) Compression set tests are intended to measure the ability of rubber compounds to retain elastic properties during prolonged action of compressive stresses. The actual stressing in service may involve the maintenance of a definite deflection, the constant application of a known load or the rapidly repeated deformation and recovery resulting from intermittent compressive forces. Though the latter dynamic stressing, like the others, produces compression set, its effects as a whole are more closely simulated by compression flexing or hysteresis tests. Therefore, compression set tests are considered to be only those involving static loading.

(*b*) These methods cover the following two test procedures using different types of load application:

Method A.—Test for compression set of vulcanized rubber under constant load.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

Method B.—Test for compression set of vulcanized rubber under constant deflection.

(*c*) The choice of method is optional but consideration should be given to the nature of the service for which correlation of test results may be sought. Notation shall be made in each test of the method actually used.

4. In case of conflict between the provisions of these methods and those of detailed specifications or methods of test for a particular material, Method B shall take precedence.

Compression Device

5. The compression devices as specified in Section 11 for Method A and in Section 20 for Method B shall be used.

Plates

6. The plates between which the test specimen is compressed shall be made of steel of sufficient thickness to withstand the compressive stresses without bending. The surfaces against which the specimen is held shall have a highly polished chromium plate finish and shall be thoroughly cleaned and wiped dry before each test.

Oven

7. Any well-designed, uniformly heated standard dry-air oven of sufficient size to hold the compression device may be used for this test. The oven shall be provided with proper temperature control to maintain the specified temperature within a permissible variation of ±1 C. (±1.8 F.). Satisfactory circulation of the air shall be secured by means of a fan. The compression device shall be centrally located in the oven and supported above the bottom of the oven by means of a wire rack mounted on wood or asbestos block so that it is heated by indirect heat.

Gages

8. A dial gage graduated in thousandths of an inch having a raised platform $\frac{3}{8}$ in. in diameter and a spherical foot $\frac{1}{4}$ in. in diameter, actuated by a load of 3 oz. ± 0.1 oz., shall be provided for measuring the thickness of the specimen before and after loading.

Standard Test Specimen

9. The standard test specimen shall be a cylindrical disk $\frac{1}{2}$ in. in thickness, cut from the vulcanized rubber part by means of a sharp, circular die having an inside diameter of 1.129 in. ±0.001 in. In cutting the specimen, the die shall be suitably rotated in a drill press or similar device and lubricated by means of soapy water so that a smooth-cut surface having square edges is obtained. The cutting pressure shall be kept sufficiently small to avoid "cupping" of the cut surface. When necessary,

the specimen thickness shall be reduced to the required $\frac{1}{2}$ in. by cutting transversely with a sharp knife and carefully buffing to size within a permissible variation of +0.005 in. and –0.025 in. The buffing shall be lightly done to avoid overheating and care must be taken to keep the circular faces parallel and at right angles to the axis of the cylinder.

Special Test Specimens

10. In routine production testing it is sometimes more convenient to cut test specimens of a different size and shape than that specified as standard. When any other size of specimen is used the test results shall not be compared with those secured using the standard test specimen. When using specimens of special sizes or shapes the results may be compared only with those from similar sizes and shapes.

Method A. Test for Compression Set Under Constant Load

Compression Device

11. The compression device shall consist of two parallel plates between which the test specimen is compressed and a calibrated loading spring, all mounted in a suitable housing having a screw mechanism for compressing the spring to apply the load and equipped with a scale graduated in hundredths of an inch for measuring the deflection of the spring. The device shall be portable, self-contained and capable of applying the desired load gradually and uniformly over the entire top and bottom surfaces of the test specimen for the duration of the test. A suitable compression device is shown in Fig. 1.

Spring

12. The loading spring shall be made of properly heat-treated spring steel with ends ground square and perpendicular to the longitudinal axis of the spring so as to help in keeping the plates parallel throughout the test. The spring shall conform to the following requirements:

(*a*) The spring shall be calibrated at room temperature (24 C. ± 5 C.) (75 F. ± 9 F.) by applying successive increments of load of 50 lb. ± 0.5 lb. and measuring the corresponding deflections in hundredths of an inch. The curve obtained by plotting the loads against the corresponding deflections shall have a slope of 4.0 ± 0.2 at 400-lb. load. The slope is obtained by dividing the difference between two loads expressed in pounds by the difference between the corresponding deflections expressed in hundredths of an inch.

NOTE.—*Example:* A spring which deflects 1.00 in. at 400 lb. and 0.75 in. at 300 lb. gives a slope of 4.0.

(*b*) The original dimensions of the spring shall not change due to fatigue by more than 0.01 in. after it has been mounted in the compression device, compressed under a 400-lb. load and heated in the oven for one week at 70 C. (158 F.). In ordinary use, a weekly check of the dimensions shall show no greater change than this over a period of 1 yr.

(*c*) The minimum load required to close the spring solid shall be 530 lb.

Original Thickness Measurement

13. The original average thickness of the test specimen shall be measured to the nearest 0.001 in. The specimen shall be placed on the small platform of the dial gage so that the pressure foot will indicate the thickness at the central portion of the top and bottom faces.

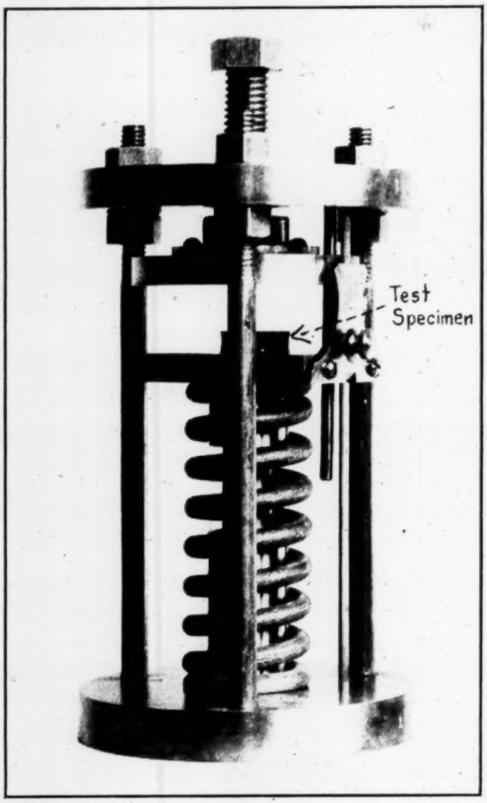

FIG. 1.—Device for Compression Set Test Under Constant Load.

Application of Load

14. The test specimen shall be assembled in the compression device using extreme care to place it exactly in the center between the plates so that tilting may be avoided. The spring shall be compressed by tightening the screw until the deflection as read from the scale is equivalent to that

shown on the calibration curve for the spring as corresponding to a load on the sample of 400 lb.

Heat Treatment

15. The assembled clamp shall be placed in the oven within 2 hr. after the completion of the assembly and shall remain there for 22 hr. in dry air at 70 C. (158 F.). At the end of the heating period the clamp shall be removed from the oven and the test specimen immediately taken from the clamp.

Cooling Period

16. While cooling, the test specimen shall rest on a wooden table top for 30 min. before the measurement of the final thickness is made.

Final Thickness Measurement

17. After the rest period, the final thickness shall be measured at the center of the test specimen using the dial gage described in Section 8.

Check Tests

18. Tests shall be run in duplicate but only one test specimen shall be tested at one time in each compression device. The duplicate test results should agree within 5.0 per cent of the values obtained.

Report

19. The report shall include the following:

(*a*) The original dimensions of the test specimen, including the original thickness, t_o.

(*b*) The actual load applied as determined from the calibration curve of the spring and the spring deflection reading.

(*c*) The thickness of the test specimen 30 min. after removal from the clamp, t_1.

(*d*) The compression set in inches and the percentage of the original thickness, calculated as follows:

$$\text{Compression set} = t_o - t_1$$

$$\text{Percentage compression set} = \frac{t_o - t_1}{t_o} \times 100$$

Method B. Compression Set Under Constant Deflection

Compression Device

20. The compression device shall consist of two or more flat steel plates between the parallel faces of which the specimens may be compressed as shown in Fig. 2. Steel spacers for the required percentage of compression given in Section 22 (*b*) shall be placed on each side of the rubber specimens to control their thickness while compressed. Four bolts shall pass through holes at the corners of the plates for compressing and holding the specimens.

Original Thickness Measurement

21. The original average thickness of the specimen shall be measured as described in Section 13.

Application of Load

22. (*a*) The test specimen shall be placed between the plates of the compression device with the spacers on each side of it. If only one specimen is being tested, it shall be placed in the center. If two specimens are being tested, three spacers shall be provided, one in the center and one outside of each specimen allowing sufficient clearance for the bulging of the rubber when compressed. The bolts shall be tightened so that the plates are drawn together uniformly until they are in contact with the spacers.

(*b*) The percentage of compression employed shall depend upon the hardness of the rubber according to the following table which is based on a value of approximately 25 in-lb. of work required to compress the standard specimen.

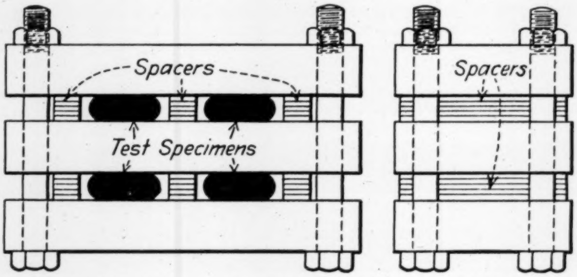

FIG. 2.—Device for Compression Set Test Under Constant Deflection.

A.S.T.M. HARDNESS NUMBER (3-LB. WEIGHT)	COMPRESSION OF RUBBER, PER CENT	SPACER THICKNESS, IN.
130 to 71	40	0.300
70 to 36	30	0.350
35 to 12	20	0.400

Heat Treatment

23. The heat treatment of the specimen shall be performed in accordance with Section 15.

Cooling Period

24. The specimen shall be cooled in accordance with Section 16.

Final Thickness Measurement

25. The final thickness of the specimen shall be measured as described in Section 17.

Check Tests

26. Tests shall be run in duplicate and the results should agree within 5.0 per cent of the values obtained.

Report

27. The report shall include the following:

(*a*) The original dimensions of the test specimen including the original thickness, t_o

(*b*) The percentage compression of the specimen actually employed.

(*c*) The thickness of the test specimen 30 min. after removal from the clamp, t_1

(*d*) The compression set in inches and the percentage of the original thickness, calculated as follows:

$$\text{Compression set} = t_o - t_1$$

$$\text{Percentage compression set} = \frac{t_o - t_1}{t_o} \times 100$$

TENTATIVE METHODS OF TEST
FOR
ABRASION RESISTANCE OF RUBBER COMPOUNDS[1]

A.S.T.M. Designation: D 394 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1934; REVISED, 1934, 1937.

Scope

1. This method is designed to measure the abrasion resistance of soft vulcanized rubber compounds, such as gum stocks, boot and shoe stocks, tread stocks, etc. No relation between this test and service performance is given or implied. The significance to be attached to the results can only be determined by each laboratory for its particular problem. It shall not be used for purchase specifications because of the difficulty of reproducing comparison standard compounds in separate laboratories. The value of having the test in standardized form for inter-comparisons and for the interpretation of results is, however, unquestioned.

Type of Materials Tested

2. Any vulcanized rubber compound, whether prepared experimentally in the laboratory, taken from process during manufacture, or cut from a finished article of commerce (providing a section of sufficient size is available), may be tested. It is assumed that the bulk of the testing will be on compounds especially designed to withstand abrasion, and that the test will be carried out with a comparison standard of the same general characteristics.

Comparison Standards

3. In order to cover the large variety of compounds that can be tested under this method, the following three standard compounds are recommended as comparison standards. The sample shall be compared with that comparison standard having physical properties (stress-strain values and hardness) most nearly matching the sample compound.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

	COMPARISON STANDARDS		
	A	*B*	*C*
	PARTS BY WEIGHT		
First latex pale crepe...........	50	......	......
No. 1 smoked sheet............	50	100	100
Zinc oxide	5	20	5
Channel carbon black	..	30	45
Stearic acid (c. p.).............	1	2	4
Medium pine tar..............	..	......	4
Mercapto-benzo-thiazole........	1	......	1
Diortho-tolyl-guanidine.........	..	1.25	......
Phenyl-beta-napthylamine......	1	1	1
Sulfur.......................	3	3.5	3
Cure.........................	60 min. at 259 F. (126 C.) (20 lb. steam)	60 min. at 287 F. (142 C.) (40 lb. steam)	70 min. at 259 F. (126 C.) (20 lb. steam)

NOTE.—It is recognized that the compounding materials shown above may vary and should be completely defined. The attention of Committee D-11 on Rubber Products is directed toward this problem in the hope of arriving at a satisfactory solution.

Preparation of Comparison Standard[1]

4. Having determined which of the three comparison standards is to be used, the following procedure shall be followed:

(*a*) *Weighing Tolerances.*—Weighing of all ingredients shall be accurate to within 0.25 per cent of the weight specified. The final weight of the mixed batch and the sum of the weights of each ingredient shall not differ by an amount exceeding 0.6 per cent for a compounded stock or 0.3 per cent for pure gum or master-batched stocks.

(*b*) *Mill Data.*—The rolls of the laboratory mill shall be 6 in. in outside diameter by 12 in. in length with 10½ in. between guides. The speed of the slow roll shall be 24 r.p.m. and the gear ratio 1.4 to 1.

(*c*) *Mill Temperature.*—The temperature of the water entering the rolls shall be maintained at 158 F. (70 C.) and the initial temperature of the rolls shall be 158 F. (70 C.). With sufficient water passing through the rolls and a uniform initial temperature, it is certain the mixing process can be made quite uniform.

(*d*) *Mill Opening.*—It is essential that the relation between batch volume and mill opening shall be regulated to develop an active bank. Since it is neither practical nor economical to standardize the size of the batch, it is recommended that mill openings be adjusted to the volume of batches as shown in the following table:

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[1] This procedure has been adapted from the "Tentative Standard Laboratory Procedure for the Preparation and Physical Testing of Rubber Samples" prepared by the Physical Testing Committee, Rubber Division, Am. Chemical Soc., *The Rubber Age* (New York), January 28, 1930.

VOLUME OF BATCH, CU. CM.	DISTANCE BETWEEN ROLLS, IN.
1200	0.170
1100	0.160
1000	0.145
900	0.130
800	0.120
700	0.110
600	0.100
500	0.085
400	0.070
300	0.055

(*e*) *Mixing Procedure.*—During breakdown, the mill opening shall be 0.055 in. until the rubber runs smooth on the roll, and then the opening shall be made to correspond to the volume of the batch in the foregoing table. The order of adding ingredients to the broken down rubber shall be as follows:

Accelerators and antioxidants
Black
Fillers
Softeners
Sulfur

The ingredients shall be incorporated as rapidly as possible and when all are in the rubber the batch shall be cut six times two-thirds of the distance across the roll, held until the bank just disappears alternating from one side to the other. Then the batch shall be cut across and rolled six times inserting the roll endwise five times and the last time it shall be inserted lengthwise. As soon as the bank is well balanced, the mill opening shall be set to give a sheet slightly greater in thickness than the test specimen after cooling and the batch shall then be cut from the rolls.

(*f*) *Storage of Mixed Batch.*—The batch shall be laid upon a suitable surface (zinc, holland, talced zinc makes no difference) until cool, after which it shall be stored 18 to 24 hr. on galvanized wire screen (6 mesh is convenient) in subdued light with free circulation of air having a relative humidity of 45 per cent at 82 F. (27.6 C.). Variation of 0 to 100 per cent relative humidity may cause a difference of 25 per cent in physical properties.

(*g*) *Preparation for Curing.*—The uncured stock shall be cut with a die having dimensions slightly less than those of the mold. Care shall be taken to have the stock so placed in the mold, that the direction of the grain will be parallel to the direction of abrasion when the vulcanized specimen is mounted in the abrader.

(*h*) *Mold Dimensions.*—Mold dimensions shall be governed by the test specimen needed for the test used.

(*i*) *Cleanliness of Molds.*—No preparation of any kind shall be used on molds to prevent sticking. The molds shall be kept clean, and shall be cleaned as soon as the cured stock tends to stick. Three cleaning materials are suggested: (*1*) ground emery and water, (*2*) buffer cloth, (*3*) whiting paste.

(*j*) *Maintenance of Curing Temperature.*—The specified curing temperature shall be interpreted as the inside temperature of the mold as determined by a thermocouple or a mercury thermometer in a mercury well in the mold. A similar steel block with a mercury well may be substituted for the mold as described in a previous report.[1] To avoid cool spots in platens due to condensation, presses with bored steel platens are recommended. If chamber type platens are used, good drainage should be insured by placing the steam outlet slightly below the bottom of the steam chamber.

(*k*) *Mold Temperature and Protection.*—The mold shall be brought to temperature in a closed press for a period of not less than 20 min. before inserting the uncured stocks. While curing, the mold shall be protected from drafts, by the use of some sort of shields or wooden ells. Protection often causes a rise of 1.8 F. (1 C.) in mold temperature.

(*l*) *Timing of Cure.*—The time of cure shall start at the time the ram pressure reaches its maximum and end at the release of the ram pressure.

(*m*) *Cooling Cured Slabs.*—The slabs shall be removed from the mold immediately after conclusion of the cure and plunged into water for at least 10 min. to cool. The water shall be changed frequently to prevent contamination and heating. When removed from the water the slabs shall be wiped dry and placed upon liner or screen for storage in subdued light.

(*n*) *Conditioning of Specimens.*—The test specimens shall be conditioned for 24 hr. in air having a relative humidity of 45 per cent at 82 F. (27.6 C.) before testing at the same temperature.

(*o*) *Preparation for Test.*—That face of the test specimen which is to be abraded shall be lightly buffed before test, except in those cases where otherwise specified.

(*p*) *Maintenance of Standards.*—It is recommended that sufficient standard compound be prepared at one time to last approximately one month. Subsequent batches of standard shall be tested for stress-strain properties and hardness and shall not vary more than 10 per cent from the original standard adopted. Its abrasion resistance shall be within 5 per cent of the batch of comparison standard being used at that time.

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[1] See Report of Physical Testing Committee, Rubber Division, Am. Chemical Soc., *Industrial and Engineering Chemistry*, Vol. 17, No. 5, p. 535, May, 1925.

Preparation of the Sample

5. (*a*) If a laboratory comparison is being made, the sample shall be prepared by the same procedure as described in Section 4 for the preparation of the comparison standard.

(*b*) If stock is taken from production, the sample shall be prepared as described in Section 4 (*f*) to (*p*), inclusive, for the preparation of the comparison standard.

(*c*) If an article of commerce is being tested, a piece somewhat larger than the test specimen required shall be cut from the sample and subsequently trimmed and buffed to size.

Method A. National Bureau of Standards Abrader[1]

Test Specimen

6. The test specimens shall be 1 in. square by $\frac{1}{4}$ in. in thickness.

Apparatus

7. The essential features of the apparatus, illustrated in Fig. 1, are as follows: The rubber-coated metal drum, *a*, is 6 in. in diameter and is rotated at 40 r.p.m. by an electric motor with a reducing mechanism, the revolutions of the drum being indicated by a counter attached to one end of the shaft. The three arms, *b*, are each pivoted at one end, and each having a weight suspended at the other end, such that a downward force of 5 lb. is exerted directly on the test specimen on the underside of the arm, *h*. The three dial gages, *c*, graduated in thousandths of an inch, fastened to a bridge so that each contacts with the corresponding arm, *b*, at a point directly over the test specimen. This bridge is hinged at one end to allow the arms to be swung back for the placing of test specimens on them. The compressed air line, *d*, is provided for keeping the abrasive surface clean. Arm stops, *e*, and an abrasive, *f*, consisting of No. $2\frac{1}{2}$ garnet paper or cloth 6 in. in width, held on the rubber-covered drum by four rubber bands. The ends of the abrasive sheet are cut at an angle of about 80 deg. and when in place have a clearance of about $\frac{1}{16}$ in. Test specimens, *g*, 1 in. square and approximately $\frac{1}{4}$ in. in thickness are cemented to small fiber disks which in turn are fastened to the weighted arms.

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[1] P. A. Sigler and W. L. Holt, "A Simple Abrasion Test Machine for Rubber," *India Rubber World*, Vol. 82, No. 5, p. 63, August, 1930.

Procedure

8. (*a*) In making a test, the specimens shall first be allowed to wear until the surface conforms to the shape of the drum. The machine shall then be stopped, the gage bridge locked in place, and all gages and the counter set at zero. The machine shall then be run until about 0.1 in. has been abraded from the standards, and the machine again stopped, and the counter and gage readings recorded. The readings shall always be taken with the drum in the same position. For routine work, these readings give the abrasion resistance in terms of "revolution per 0.1 in. wear."

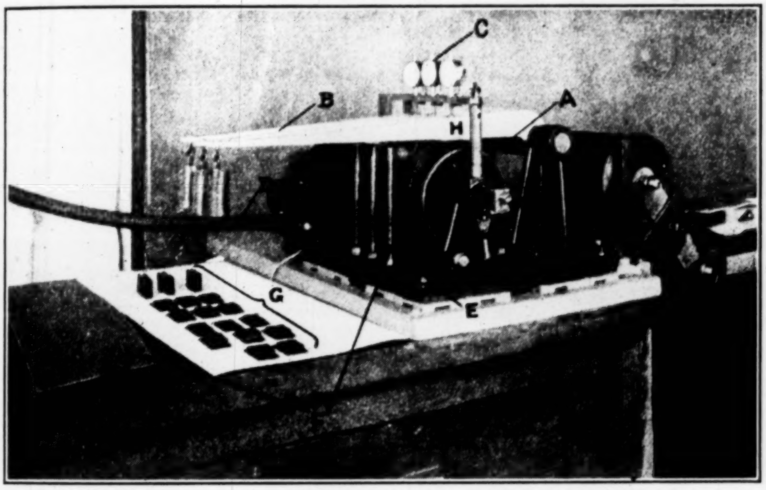

FIG. 1.—National Bureau of Standards Abrader.

(*b*) For purposes of conforming to this method, the weight of the test specimen before and after abrading may be measured and the volume loss calculated.

(*c*) From the loss in weight, the volume loss and abrasion resistance shall be calculated in accordance with Section 24.

Method B. E. I. du Pont de Nemours and Co. Abrader[1]

Test Specimen

9. The test specimens shall be 2 cm. square by 1 cm. in thickness.

Apparatus

10. The essential features of the apparatus, illustrated in Fig. 2, are as follows: A disk which carries an abrasive surface is mounted

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[1] Ira Williams, "Measurement of Abrasion Resistance of Rubber," *Industrial and Engineering Chemistry*, Vol. 19, No. 6, p. 674, June, 1927.

on a hollow shaft and rotates counter-clockwise in a vertical plane at a speed of 37 r.p.m. Two test specimens are mounted on the inside of a bar, one placed at each end so that their centers are a distance of 5 in. apart. This bar is attached permanently to a rod (axis) which extends through the hollow shaft carrying the abrasion disk. A weight attached to the end of this rod by means of a cable and acting over a pulley holds the test specimens against the abrasive. A lever arm is attached to the end of the bar which holds the test specimens. The other end of this lever arm carries a bucket, the weight of which is

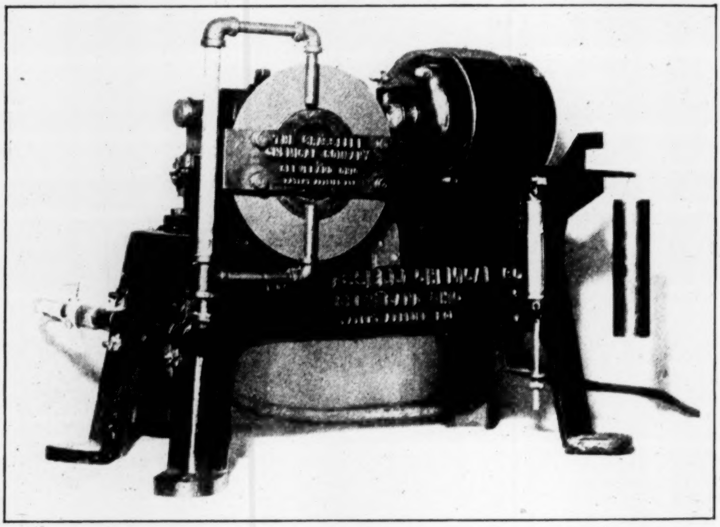

Fig. 2.—E. I. du Pont de Nemours and Co. Abrader.

adjusted with shot, just enough to prevent rotation. A vernier spring balance is attached to the lever arm for final adjustment of the load. The abrasive generally used is No. 0 emery paper. The abrasive surface is cleaned by means of air jets.

Procedure

11. (*a*) Two test specimens shall be inserted in the clamps of the balance lever and the clamps then tightened uniformly, care being taken to avoid distorting the surface to be abraded. The balance lever shall be placed in position with its axis rod extending through the hollow shaft which supports the abrasive disk.

The same procedure shall be followed inserting two test specimens of the comparison standard in the clamps. The 3.62-kg. weight shall be connected to the end of the axis rod, leveling the cable over the grooved pulley, thus holding the test specimens evenly and firmly against the abrasive disk. The balance weight (bucket) shall be attached to the end of the lever arm and the spring balance connected to the lever arm (for most tests the total weight of the bucket and shot will be 500 g.).

(*b*) The surface of the abrasive disk shall first be cleaned by applying 20-lb. air pressure to them through the jets. The air shall be filtered to remove oil, water and dirt. Any evidence of these appearing on the abrasive disk shall be cause for repeating the test. The machine shall be run until the test specimens are seated evenly, the specimens then removed and weighed. The test specimens shall then be replaced in exactly the same position as before and run for 20 min., adjusting the load applied by means of the spring balance to offset rotation of the balance lever. The lever arm will fluctuate slightly between the stops provided on the machine, but should not touch the stops. At the completion of the test, the specimens shall be removed and weighed.

(*c*) From the loss in weight, the volume loss and abrasion resistance shall be calculated in accordance with Section 24.

Method C. The B. F. Goodrich Co. Abrader[1]

Test Specimen

12. The test specimens shall be rings 1.5 in. in outside diameter by 1 in. in inside diameter and at least $\frac{3}{32}$ in. in thickness.

Apparatus

13. The essential features of the apparatus, illustrated in Fig. 3, are as follows: A steel mandrel 1 in. in diameter and 3 in. in length, over-all, on which the ring test specimens are mounted, and a cylindrical can 3 in. in diameter and 9 in. in depth filled with No. 6 carborundum. In addition to the ring specimens, the mandrel carries hardened steel spacer rings 1 in. in inside diameter by $1\frac{5}{32}$ in. in outside diameter by $\frac{3}{16}$ in. in thickness, which are knurled on the faces adjacent to the specimens. These serve to keep the specimens separated and to clamp them firmly when assembly is completed by tightening a nut on the end of the mandrel. The mandrel is fastened to a vertical shaft in such a way that the sample rings are revolved at fixed depths in the can filled with carborundum. The mandrel is

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[1] W. W. Evans, "An Apparatus and Method for Determination of Resistance to Abrasion of Rubber Products," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 517 (1923).

driven by a reversible motor at a speed of 120 r.p.m. The can has an 8-mesh screen on the bottom to permit broken grains and rubber dust to escape. The abrasive mixture is always screened after use, one part of new abrasive being added to four parts of old before screening. This proportion maintains a uniform supply of abrasive.

Procedure

14. Test specimens of both the sample and the comparison standard shall be weighed and placed on the mandrel, alternating the sample and the comparison standard. All specimens shall be kept separated by hardened steel spacers. The mandrel shall be mounted on the shaft, the can placed around it and the carborundum filled in around the rings. The motor shall then be started, the time noted, and the specimens attached to the mandrel allowed to revolve in the carborundum. When the test is half finished, the mandrel shall be turned upside down and the motor reversed in order to

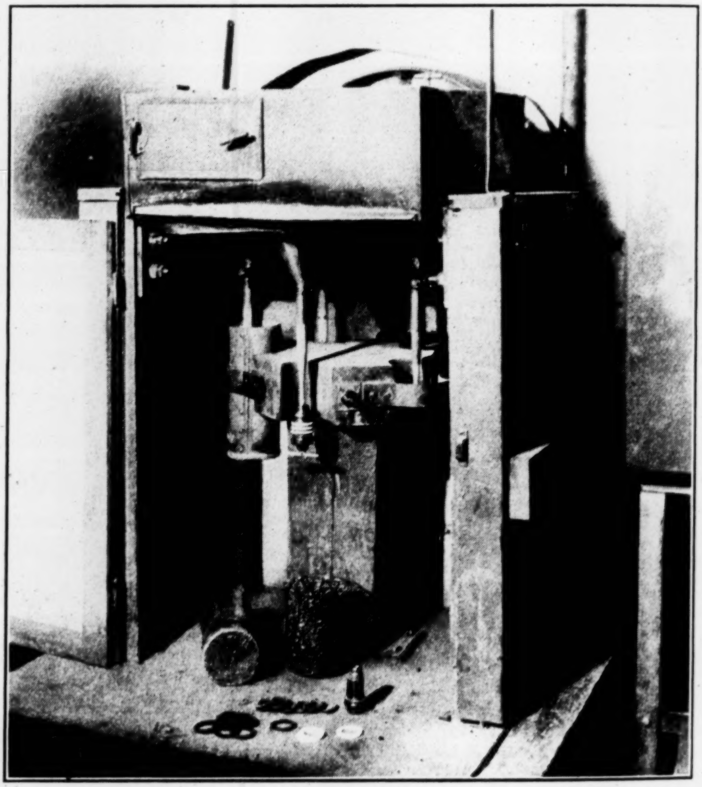

FIG. 3.—The B. F. Goodrich Co. Abrader.

equalize the abrasion on the test specimens. The duration of the test depends on the rate of wear of the material being tested. In general, the test shall be continued until there is at least 100 mg. loss in weight on each of the specimens. At conclusion of the test, the specimens shall be weighed and the loss in weight obtained by difference.

NOTE.—*Example:* A compound of quality similar to that in rubber heels may require a wearing period of approximately 2 hr., while a high grade pure rubber compound such as inner tube stock may require 6 hr. for satisfactory results.

(*d*) From the loss in weight, the volume loss and abrasion resistance shall be calculated in accordance with Section 24.

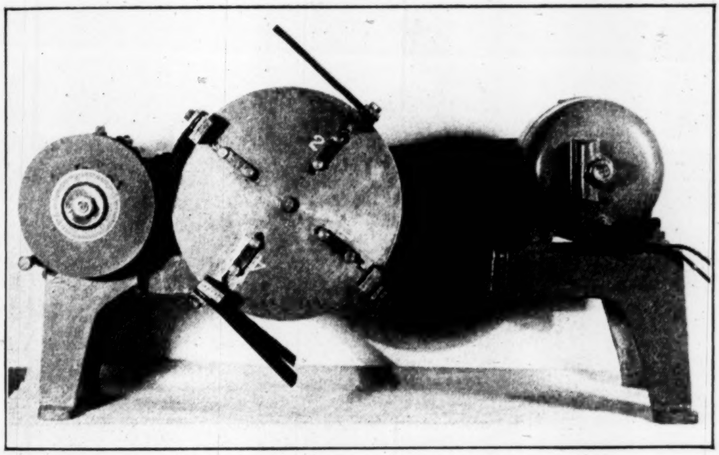

FIG. 4.—Kelly-Springfield Abrader.

Method D. Kelly-Springfield Abrader[1]

Test Specimen

15. The test specimens shall be mold-cured slabs $2\frac{1}{2}$ by $5\frac{3}{16}$ in. by $\frac{1}{4}$ in. in thickness with a very slight offset under the specified thickness at one end provided for mounting the specimen.

Apparatus

16. The essential features of the apparatus, illustrated in Fig. 4, are as follows: An abrasive wheel (Note) $1\frac{1}{2}$ in. in width and 6 in. in diameter which travels against the face of the specimen at a rate of 2386 ft. per minute. For mounting the specimens tangentially to the 10-in. wheel which revolves at 70 r.p.m., plates of spring steel $\frac{1}{2}$ in. shorter than the specimen shall be used.

NOTE.—Abrasive Wheel No. 1960-J made by the Norton Co., Worcester, Mass., or its equivalent, is satisfactory.

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[1] A. F. Hardman, W. L. Mackinnon and S. M. Jones, "The Kelly Abrasion Machine," *The Rubber Age*, Vol. 28, No. 9, p. 463, February 10, 1931.

Procedure

17. (*a*) The specimens shall be trimmed and then weighed to the nearest centigram before mounting on the spring plates. Three different compounds and the comparison standard shall be tested on four different runs of the machine. The position of each specimen shall be changed after each run and the results averaged. All positions of the mounted specimen must be kept filled during each test, if only with blanks, to protect the spring plates from the abrasive wheel. Each test shall be run for 675 revolutions of the test specimen, and the specimen shall be again weighed after cleaning thoroughly.

(*b*) From the loss in weight, the volume loss and abrasion resistance shall be calculated in accordance with Section 24.

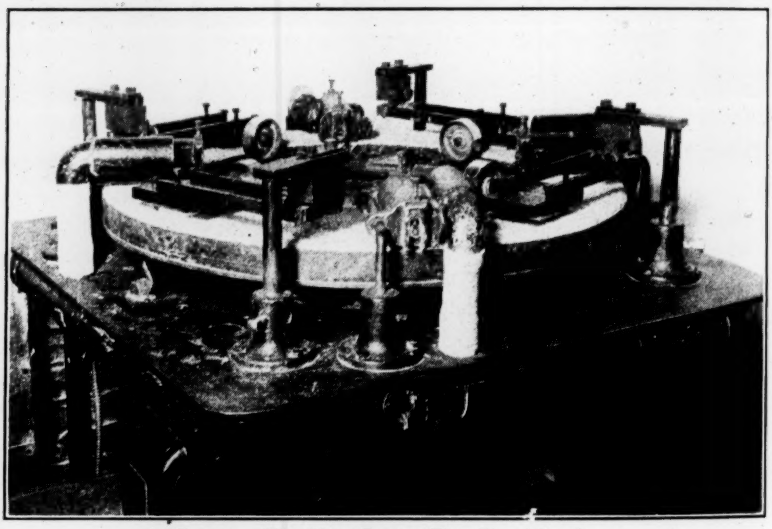

FIG. 5.—The New Jersey Zinc Co. Abrader.

Method E. The New Jersey Zinc Co. Abrader[1]

Test Specimen

18. The test specimens shall be blocks of rubber compound 2 by 2 in. by 0.25 in. in thickness. For convenience, a mold 2 by 7 in. may be used, providing three test specimens.

Apparatus

19. The essential features of this testing apparatus, illustrated in Fig. 5, are as follows: The horizontal abrasion track is 30 in. in

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[1] H. A. Depew, "An Explanation of Some of the Difficulties in Abrasion Testing of Rubber," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 871 (1928).

outside diameter and 24 in. in inside diameter, the abrasive surface being feldspar embedded in concrete. The track is revolved at a speed of 30 r.p.m. and the dust is removed by brushes and suction. The machine is provided with four arms which bear on the abrasive track and six lifts raise and lower the 2 by 2 by 0.25-in. specimens to give cutting and tearing action.

Procedure

20. (*a*) After dieing out the test specimen to the 2 by 2 in. by 0.25-in. size, it shall be buffed lightly on a large surface. A holder, made up of hard rubber faced with a soft rubber surface on one side shall be used, to which the test specimen shall be cemented. The mounted samples shall be placed in a clamp under pressure for approximately 12 hr. before testing.

(*b*) Four test specimens shall be placed into the slots of the four arms of the abrasion machine. Three specimens of unknown value shall be tested along with a specified comparison standard. The tracks shall be revolved at 30 r.p.m., the usual test being 1000 revolutions of the track. If a specimen should wear through before the 1000 revolutions are completed, a warning is provided by the soft blue rubber facing on the hard rubber holder. In such a case, the calculations shall be based on the actual number of revolutions of the track to failure.

(*c*) The track shall be kept free from the abraded rubber dust by means of air suction and revolving brushes at each of the four arms.

(*d*) The samples shall be weighed before and after abrasion. From the loss in weight, the volume loss and abrasion resistance shall be calculated in accordance with Section 24.

Method F. United States Rubber Co. Abrader[1]

Test Specimen

21. The test specimens shall be slabs 2 by 3 by $\frac{3}{8}$ in. or 2 by 5 by $\frac{3}{8}$ in. to fit the standard machine.

Apparatus

22. The essential features of the apparatus, illustrated in Fig. 6, are as follows: A rotating wheel 3 in. in diameter with the outer rim 1 in. in width and covered with an abrasive cloth (metallic cloth, 60 grit). A specimen carrier moves the test specimen held in a suitable grip against this wheel, the wheel rotating against the direction of the movement of the table. The rotation of the wheel is reversed at the time the movement of the specimen table is reversed. Mechanical

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[1] Described in catalogue of Henry L. Scott Co., Providence, R. I.

means are provided to raise the wheel from the work as it passes from specimen to specimen. An exhaust blower is provided to remove all dust from the wheel and specimens, and a small revolving brush to clean the wheel thoroughly as it is raised between the grinding of each specimen. This will insure against the carrying of particles from one specimen to the next. A balance arm is provided for the purpose of regulating the pressure of the wheel on the specimens.

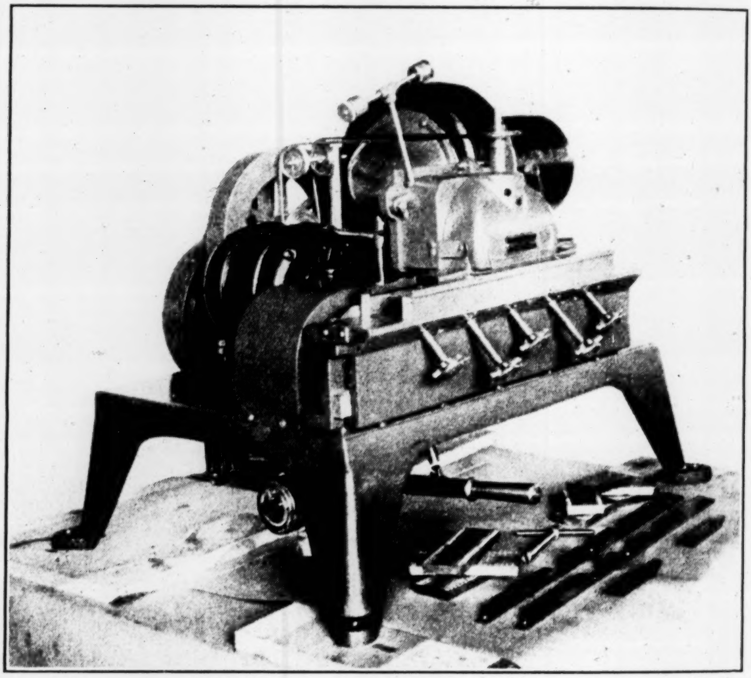

FIG. 6.—United States Rubber Co. Abrader.

The machine is equipped with a counter which registers the number of cycles or applications of the abrasive wheel to each specimen.

Procedure

23. (*a*) After the specimens to be tested have been weighed, they shall be placed in the machine and firmly fastened in place, care being taken that the faces to be abraded are all in the same plane and exactly flush with the restraining edges of the traveling table. A fresh piece of abrasive cloth shall be used for each test.

(*b*) The machine should be at the end of a cycle so that when started, each specimen will receive the same number of applications of the abrasive wheel provided that at the conclusion of the test the machine is again stopped at the end of a cycle. The machine is geared to make nine cycles per minute and the length of the test to be made will necessarily depend upon the type of stock being tested. The test shall be run long enough to remove sufficient quantity of the specimen by abrasion to reduce the percentage of error to a minimum. A general rule which should apply requires that the test run until the specimen has been abraded to such a depth that there is still 0.15 in. (0.039 cm.) of material at the point of the deepest cut. The test shall be stopped at a convenient time when abrasion has proceeded to practically this point, at an even number of cycles to facilitate the calculation of results to a common basis for comparison. The test specimens shall then be removed and weighed after brushing them off to remove any loose particles which might remain adhering to the specimens.

(*c*) From the loss in weight, the volume loss and abrasion resistance shall be calculated in accordance with Section 24.

Calculation of Results

24. (*a*) In each of the six alternative methods of test described in Sections 6 to 23 the loss in weight of the test specimen shall be accurately measured to within ±1 mg., unless otherwise specified.

(*b*) The specific gravity of the stock shall be determined to the second decimal place. The volume loss shall be calculated by dividing the loss in weight by the specific gravity.

(*c*) The abrasion resistance shall be calculated by dividing the volume loss of the standard by the volume loss of the sample and multiplying by 100.

(*d*) The results shall be expressed as a percentage of the comparison standard specified.

NOTE.—*Example:* 110 per cent of A.S.T.M. Comparison Standard ***A***.

TENTATIVE METHODS OF TEST

FOR

CHANGES IN PROPERTIES OF RUBBER AND RUBBER-LIKE MATERIALS IN LIQUIDS[1]

A.S.T.M. Designation: D 471 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.[2]

Scope

1. (*a*) These methods are intended for use in estimating the comparative ability of rubber and rubber-like compositions to withstand the effect of liquids. Owing to the wide variations often present in service conditions, no direct correlation between these accelerated tests and service performance may be given or implied. However, the methods yield comparative data on which to base judgment as to service quality and are especially useful in research and development work. They are designed for testing any vulcanized rubber or rubber-like composition in the form of standard laboratory test sheets and should not be used for samples cut from a finished article of commerce. It is assumed that the tests will be used principally for intercomparisons between compounds rather than for an estimation of service performance of manufactured products.

(*b*) Except as may be otherwise specified in these methods, the requirements of the Tentative Methods of Physical Testing of Rubber Products (General Requirements) (A.S.T.M. Designation: D 15 – 35 T) of the American Society for Testing Materials[3] shall be complied with and are made a part of these methods.

Types of Test

2. These tests consist of exposing test specimens to the influence of liquids under definite conditions of temperature and time for the purpose of measuring the resulting deterioration. The type of test varies depending upon the conditions of test and the nature of the liquid used. In any case, the deterioration is measured by the change in physical properties before and after immersion in the liquid. The physical properties used are changes

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

[2] Accepted for publication as tentative by Committee E-10 on Standards, August 26, 1937.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1153 (1935); also 1937 Book of A.S.T.M. Tentative Standards, p. 1260.

in weight, volume, tensile strength, and ultimate elongation, as well as visual and manual inspection.

Test Conditions

3. (*a*) *Temperature.*—A suitable test temperature shall be chosen depending upon the conditions of the expected service. It shall be equal to or higher than the anticipated service temperature. In comparative tests, identical temperatures shall be employed. Use of one of the following temperatures is recommended: 25 C. ± 5 C.; 70 C. ± 1 C.; 100 C. ± 1 C.

(*b*) *Periods of Immersion.*—Choice of the period of immersion to be used in any given test depends upon the nature of the composition, the temperature of test, and the liquid employed. Periods of 24 hr., 48 hr., 7 days, and 30 days are suggested as suitable but it is recommended that determinations be made and recorded after several periods of immersion in order to obtain information concerning the rate of deterioration.

(*c*) *Liquids.*—Tests shall be made using the liquid with which the test material will come in contact in service. For comparative tests with liquids of unknown or questionable composition, samples from the same drum or shipment of liquid shall be used. This is of great importance when using such liquids as gasoline, kerosine, mineral oil, and gas house drip oil.

(*d*) All immersion tests shall be made in the absence of light.

Apparatus

4. The following apparatus shall be used for immersing each test specimen:

(*a*) *For Volatile Liquids.*—A glass test tube having an outside diameter of 38 mm. and an over-all length of 300 mm., fitted tightly with a cork stopper and a reflux condenser. A suitable bumper such as glass beads shall be used in the liquid in the test tube.

(*b*) *For Non-Volatile Liquids.*—A glass test tube having an outside diameter of 38 mm., and an over-all length of 200 mm., fitted loosely with a cork stopper. A glass test tube having an outside diameter of 38 mm., and an over-all length of 300 mm., may be used.

Test Specimens

5. (*a*) The test specimens shall be cut longitudinally from vulcanized sheets prepared as described in Sections 4 to 6 of the Tentative Methods of Physical Testing of Rubber Products (General Requirements) (A.S.T.M. Designation: D 15 – 35 T) of the American Society for Testing Materials.[1]

(*b*) For the determination of changes in weight or volume, and for visual or manual inspection, rectangular test specimens having dimensions of 1 by 2 by 0.075 in. shall be used.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1153 (1935); also 1937 Book of A.S.T.M. Tentative Standards, p. 1260.

(*c*) For immersion in connection with the determination of changes in tensile strength and ultimate elongation, rectangular test specimens having dimensions of 1 by 5 by 0.075 in. shall be used.

(*d*) At least three test specimens shall be used for each determination and the results averaged.

INCREASE IN WEIGHT

Procedure

6. The test specimen shall be weighed in air to the nearest milligram and placed in a test tube as described in Section 4 (*a*) or (*b*), depending on the type of liquid to be used in the test. To this test tube shall be added 100 ml. of test liquid. If a non-volatile liquid is being used, the test tube may then be placed in a thermostatically controlled air oven for the required length of time. However, if a volatile liquid is being used, the test tube shall be fitted with a reflux condenser as described in Section 4 (*a*) and placed in a liquid bath thermostatically controlled at the desired temperature of test. In either case a control test tube containing the test liquid or any other suitable medium and a precision thermometer shall be used along with the test tubes containing the test specimens. After the immersion test has proceeded the required length of time, the test specimen shall be removed, dipped quickly into 95 per cent ethyl alcohol, blotted lightly with filter paper, placed immediately in a tared, stoppered, weighing bottle and the weight after test determined. After weighing, the test specimen shall be again immersed in the test liquid if data is desired on the progressive changes which occur with increasing time of immersion. It is important that each manipulation take place promptly with the least possible elapse of time. No more than 30 sec. shall be allowed for each step except the actual weighing in the tared, stoppered, weighing bottle.

INCREASE IN VOLUME

Procedure

7. (*a*) *Water Displacement Method for Water Insoluble Liquids and Mixed Liquids.*—The initial water displacement of the specimen shall be determined by weighing it in air and in distilled water at room temperature, the difference being the weight of the water displaced by the specimen. The specimen shall then be dipped quickly into 95 per cent ethyl alcohol, blotted dry with filter paper, and placed in the immersion apparatus as described in Section 4 (*a*) or (*b*). One hundred milliliters of liquid shall then be added to the test tube and the immersion test carried out as described in Section 6. At the end of the required length of immersion time, the specimen shall be removed from the test tube and dipped quickly into 95 per cent ethyl alcohol at room temperature, blotted lightly with filter paper, placed in a tared weighing bottle and weighed. It shall then be removed from the bottle and weighed in distilled water in im-

mediate consecutive procedure to determine the water displacement after test. If the immersion has been at an elevated temperature, the test specimen should be cooled to room temperature at the end of the immersion period by being transferred to a cool, clean portion of the test liquid prior to being dipped in alcohol. It is important that all air bubbles clinging to the test specimens be removed during the weighing in distilled water. When data is desired on the progressive changes occurring with increasing time of immersion, the test specimen shall be immediately removed from the water after making this determination, dipped quickly into 95 per cent ethyl alcohol at room temperature, blotted lightly with filter paper to remove the water and alcohol, and replaced in the test liquid with the least possible lapse of time.

(*b*) *Specific Gravity Method for Water Soluble Liquids of Known Composition.*—This method is not satisfactory for mixed liquids because the specific gravity of the absorbed liquid may differ from that of the bulk. The same procedure shall be used as for the water displacement method described in Paragraph (*a*), except that after blotting the test specimen with filter paper and weighing, it is not removed from the bottle nor weighed in water.

CHANGES IN TENSILE STRENGTH AND ELONGATION

Procedure

8. The tension testing of the specimens in this method shall be as described in the Tentative Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412 – 36 T) of the American Society for Testing Materials,[1] excepting that the dumbbells shall be the 1 by $\frac{1}{4}$-in. shape (Section 8, Fig. 3, Die C) and that the pressure foot of the micrometer (Section 10) used for thickness measurement shall exert a total force of 3.0 oz. The original tensile strength and elongation shall be determined using duplicate specimens to those which are to be immersed in liquid. The rectangular specimens, prescribed in Section 5 (*c*), shall be placed in test tubes as described either in Section 4 (*a*) or (*b*) and 150 ml. of liquid added to each. The immersion test shall then be carried out as described in Section 7 (*a*) except that after blotting the test specimens with filter paper the procedure varies according to whether it is required to determine the deterioration immediately or after recovery. For testing the immediate deteriorated properties, dumbbell tension test specimens shall be died out, gaged and tested immediately. For the recovered properties the specimens shall be suspended in air, protected from light and draft, and held at room temperature for 24 hr., after which dumbbell specimens shall be died out, gaged and tested. The procedure actually employed shall be noted in the report.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1105 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1266.

CALCULATIONS AND EXPRESSION OF RESULTS

Calculations

9. (*a*) The increase in weight (Section 6) shall be calculated as follows:

$$\text{Percentage Increase in Weight} = \frac{\text{Weight After Test} - \text{Initial Weight}}{\text{Initial Weight}} \times 100$$

(*b*) The increase in volume shall be calculated as follows:

Water Displacement Method (Section 7 (a)):

$$\text{Percentage Increase in Volume} = \frac{\text{Water Displacement After Test} - \text{Initial Water Displacement}}{\text{Initial Water Displacement}} \times 100$$

Specific Gravity Method (Section 7 (b)):

$$\text{Percentage Increase in Volume} = \frac{\text{Percentage Increase in Weight} \times \text{Specific Gravity of Sample}}{\text{Specific Gravity of Liquid}}$$

(*c*) The deterioration in tensile strength and ultimate elongation (Section 8) shown by the difference between the determinations before and after immersion shall be expressed as a percentage of the original property.

Report

10. The report shall include the following:

(*a*) The results calculated in accordance with Section 9,
(*b*) All observed and recorded data on which the calculations are based,
(*c*) The exposure period,
(*d*) The temperature of exposure,
(*e*) The liquid used,
(*f*) Statement of condition of exposed specimens from visual and manual examination,
(*g*) The duration, temperature, and date of vulcanization of the test specimens, and
(*h*) The dates of the various periods of exposures.

TENTATIVE METHODS OF TEST
FOR
RUBBER HOSE[1]

A.S.T.M. Designation: D 380 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.[2]

Scope

1. (*a*) These methods are intended for use in the examination and test of all types and constructions of rubber hose. The term "rubber" as herein used includes synthetic compounds as well as compounds of natural rubber.

(*b*) Except as otherwise specified in these methods for rubber hose, the following methods of test of the American Society for Testing Materials, applicable in general to vulcanized rubber, shall be complied with as required and are hereby made a part of these methods:

General Physical Test Requirements.—Tentative Methods of Physical Testing of Rubber Products (General Requirements) (A.S.T.M. Designation: D 15 - 35 T).[3]

Tension Test.—Tentative Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412 - 36 T).[4]

Aging Test.—Tentative Methods of Test for Accelerated Aging of Vulcanized Rubber (A.S.T.M. Designation: D 428 - 36 T).[4]

Adhesion Test.—Tentative Methods of Test for Adhesion of Vulcanized Rubber (Friction Test) (A.S.T.M. Designation: D 413 - 36 T).[4]

(*c*) In case of conflict between the provisions of these methods and those of detailed specifications or methods of test for a particular hose, the latter shall take precedence.

SAMPLING

Sampling

2. (*a*) A sample of sufficient length for proper performance of the required tests shall be cut from the hose when possible without impairing its use and the pieces of hose from which samples have thus been cut shall be accepted by the purchaser as full length, provided the hose meets the

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1153 (1935); also 1937 Book of A.S.T.M. Tentative Standards, p. 1260.

[4] *Proceedings*, Am. Soc. Testing Mat., Vol. 36, Part I, pp. 1105, 1113, 1120 (1936); also 1937 Book of A.S.T.M. Tentative Standards, pp. 1266, 1274, 1286.

specified requirements. In general, the amount required and the frequency of sampling for each size and type of hose shall be one 30-in. sample from each 500 ft., or fraction thereof, of hose over $1\frac{1}{4}$ in. in nominal diameter and one 36-in. sample from each 500 ft., or fraction thereof, of hose $1\frac{1}{4}$ in. or under in nominal diameter.

(*b*) Where the construction of the hose does not permit the cutting of samples as described in Paragraph (*a*) without impairment of use, as in wire inserted hose, dredging sleeves, etc., a separate length having the same specifications shall be provided, either as an additional length or as a prolongation built on to the hose. Wire shall be omitted from the prolongation or sample length unless the detailed physical tests include requirements that measure the effect of the wire or metal elements in the construction. Additional lengths taken for purpose of tests shall be paid for by the purchaser, provided they conform to the specified requirements.

(*c*) The sampling of cotton-jacketed rubber-lined hose shall be in accordance with detailed specifications for this material.

MEASUREMENTS OF SIZE AND THICKNESS

Measurement of Hose

3. (*a*) *Length.*—The length shall be determined by means of a steel tape or calibrated measuring device and shall not include the length of fittings except when specifically so requested and recorded. In the case of cotton-jacketed rubber-lined hose, the length shall be determined under a 10-lb. internal pressure.

(*b*) *Outside Diameter.*—The outside diameter shall be determined by calculation from measurement of the outside circumference. A tape graduated to read the diameter directly may be used.

(*c*) *Inside Diameter.*—The inside diameter shall be measured with a plug gage except that on hose larger than 3 in. in nominal diameter, a steel scale or caliper may be used.

Thickness of Tube, Cover or Other Rubber Elements

4. (*a*) The thickness of a rubber element shall be determined by means of a micrometer graduated to thousandths of an inch and having a presser foot 0.25 in. ± 0.01 in. in diameter, exerting a total force of 9 oz. ± 0.1 oz. The load shall be applied by means of a weight.

(*b*) A rubber element adjoining a braid or helical wound reinforcing member of any material shall have the thickness taken as the average between two measurements of thickness, one of which shall be obtained using the unbuffed specimen and the other using a specimen which is buffed just sufficiently to remove the braid or helical corrugations.

(*c*) A rubber element adjoining a seamless woven jacket of any material shall have the thickness taken on a specimen which is buffed just sufficiently to remove the corrugations caused by the woven jacket. Where backing is used on a rubber element, the backing shall be removed before thickness measurements are taken.

(*d*) A rubber element adjoining flexible metal convoluted lining shall have the thickness determined at the valleys formed by the tops of the metal convolutions. The opposite side of the rubber element, adjacent to other reinforcing members, shall be prepared for thickness measurement as specified in the other paragraphs of this section, depending upon the type of reinforcing member.

(*e*) A rubber element adjoining reinforcing fabric woven prior to assembly in the hose, shall have the thickness determined on a specimen which is unbuffed.

TEST CONDITIONS AND NUMBER OF TESTS

Test Conditions

5. The temperature of the testing room shall be between 70 and 90 F. (21 and 32 C.). The samples to be tested shall be kept in this room for at least 30 min. previous to the time of testing.

Number of Tests

6. (*a*) When minimum requirements are established, one specimen shall be tested for each physical characteristic required in the detailed specifications for a particular hose except in the immersion tests given in Sections 20 to 23, inclusive. If the results do not meet the specified requirements, two additional specimens shall be prepared from the original sample and tested. Should the results of either of these tests be below the specified requirements, the sample shall be considered to have failed to meet the specifications.

(*b*) If minimum requirements are not established, at least three specimens shall be tested and the results averaged.

TENSILE STRENGTH AND ELONGATION OF RUBBER

Tension Test Apparatus

7. (*a*) *Testing Machine.*—Tensile strength and elongation tests shall be made on apparatus accurate within ± 1 per cent and provided with an accurate device for indicating the actual maximum load at rupture. The preferred type of apparatus is a power-driven machine of the inclination-balance or pendulum type, but a spring balance accurately calibrated may be used.

(*b*) *Grips for Testing.*—The grips for holding the test specimen shall be such that they tighten automatically, exerting a uniform pressure, proportional to the applied tension, across the full width of the enlarged end of the test specimen. The moving grip shall move at a constant speed of 20 in. per min.

Tension Test Specimen

8. (*a*) *Separation of Rubber Parts.*—Sufficient length shall be cut from the hose sample to provide longitudinal tension test specimens from hose 1¼ in. and under in nominal diameter and transverse test specimens on larger sizes. The rubber parts to be tested shall be separated from the balance of the hose without the use of gasoline, if possible. If it is necessary to employ gasoline to effect the separation, it shall be straight run gasoline of the quality known as aviation grade and shall have the following characteristics:

Specific gravity at 60 F	0.685 to 0.705
Distillation limits:	
Initial boiling point	100 to 120 F.
10 per cent evaporated	128 to 143 F.
20 per cent evaporated	140 to 155 F.
30 per cent evaporated	150 to 164 F.
40 per cent evaporated	156 to 174 F.
50 per cent evaporated	164 to 184 F.
60 per cent evaporated	173 to 194 F.
70 per cent evaporated	182 to 207 F.
80 per cent evaporated	193 to 223 F.
90 per cent evaporated	209 to 245 F.
95 per cent evaporated	224 to 260 F.
Dry	250 to 286 F.
Recovery, min	97 per cent

The gasoline shall contain no lead compounds and shall not leave an appreciable amount of oily residue when evaporated. After use of gasoline, the test specimen shall be allowed to rest at least an hour before testing.

(*b*) *Preparation of Specimen.*—The test specimen shall be stamped out from the separated rubber part with dies conforming to Fig. 1 *A*, *B*, or *C*. In no case shall the thickness of the test specimens exceed ⅛ in. Test specimens shall be free from marked irregularities of thickness.

(*c*) *Buffing.*—The specimen shall be buffed, but only to the extent necessary to remove surface corrugations, irregularities, or excess thickness, and to insure smooth faces. Buffing shall be done without excessive heating and the test specimen shall be allowed to rest after buffing for at least 10 min. before being tested.

(*d*) *Marking.*—Parallel gage lines 1 to 2 in. apart, depending upon the die used, shall be marked on the test specimen at right angles to the longitudinal axis by means of an accurate stamp or bench marker. Care must be taken to make fine lines and to avoid injuring the specimen.

(*e*) *Measurement of Test Specimen.*—The thickness of the test specimen shall be the minimum thickness found between the marks on the constricted portion, using the standard micrometer described in Section 4 (*a*).

Tension Test Procedure

9. (*a*) The test specimen shall be placed in the jaws. The jaws shall be separated at the specified rate of speed until rupture takes place. The

distance between the centers of the gage marks noted at the instant of rupture shall be measured using a rule or tape graduated to eighths of an inch or smaller divisions.

(*b*) Test specimens that break outside of the gage marks or are found to be imperfect shall be discarded and retests shall be made.

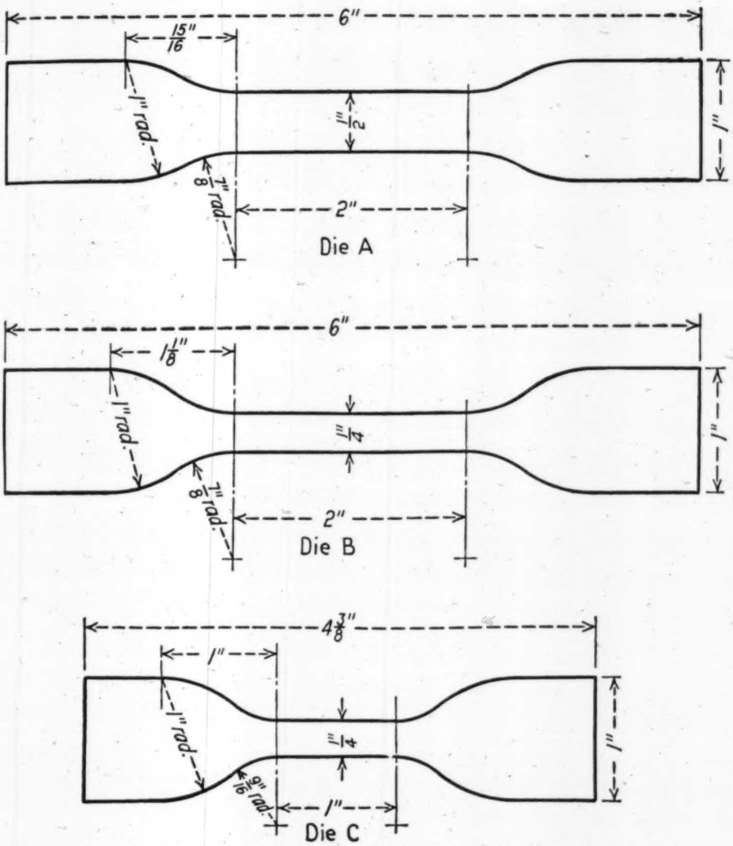

FIG. 1.—Tensile Strength and Elongation Test Specimens.

Die B shall be used when the size of the sample and the tension and stretch limits of the testing machine permit, with exceptions as explained below. If the tension limit is too low for the machine, use Die A. If the stretch is too high for the machine, use Die C. Die C shall also be used for samples which are too small for A or B.

(*c*) The tensile strength in pounds per square inch shall be calculated from the breaking load indicated by the machine and the original cross-sectional dimensions of the test specimen.

(*d*) The elongation shall be the difference between the original gage length and the total distance between the gage marks at the time of rupture as measured in Paragraph (*a*) and shall be expressed in percentage of the original gage length.

PERMANENT SET[1]

Permanent Set Test

10. The specimen for the permanent set test shall be prepared as for determining tensile strength (Section 8) and placed in a suitable stretching device. The grips shall conform to the requirements specified in Section 7 (*b*). The specimen shall be stretched to an amount equal to three-fourths of the ultimate elongation specified, held for 2 min., released, and the distance between the centers of the gage marks measured at the expiration of 2 min. The difference between this measurement and the original gage length divided by the original gage length and expressed as a percentage shall be recorded as the permanent set. If no ultimate elongation is specified, the specimen shall be stretched an amount equal to three-fourths of the elongation determined under Section 9.

ADHESION TESTS

Adhesion Test Specimens

11. (*a*) From cotton-jacketed rubber-lined hose adhesion test specimens shall be cut transversely from the samples in rings 2 in. in width. These rings shall then be opened at the lap and laid out flat so as to give rectangular specimens of length equal to the full circumference of the hose. On each specimen a central strip of lining $1\frac{1}{2}$ in. in width shall be cut accurately, the cut extending through the lining but not entirely through the cotton jacket. These strips shall be separated from the jacket at one end for a distance of about $1\frac{1}{2}$ in.

(*b*) From other types of hose the adhesion test specimens shall be cut transversely from the samples in rings 1 in. in width using a sharp tool which will leave clean edges. On hose $\frac{1}{4}$ to 4 in., inclusive, in internal diameter the specimens shall be tested in ring form and shall be referred to as "ring specimens." For hose over 4 in. in internal diameter the rings shall be cut through at the point where the outside ply of fabric ends, and opened to form strips which shall be referred to as "strip specimens."

(*c*) Adhesion specimens from hose less than $\frac{1}{4}$ in. in internal diameter shall be cut longitudinally from the hose. Since the width of these specimens will be less than 1 in., the adhesion shall be determined on the contact width of the test specimen.

(*d*) In hose constructions having braided wire or woven wire filler reinforcing members, it is impracticable to prepare adhesion test specimens except for determining cover adhesion.

Adhesion Test Procedure

12. (*a*) *Ring Specimens.*—The ring specimen shall be placed on a mandrel having a diameter equal to the nominal diameter of the hose.

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[1] The amount of permanent set is considered by many rubber technologists as having no relation to the service value of rubber compounds except for some specialized requirements. The permanent set test is, therefore, not recommended for general use.

The parts to be tested shall be separated sufficiently by hand to permit attaching the jaws of a testing clamp. With the mandrel supported so that it may revolve freely with minimum friction, the prescribed weight shall be attached to the clamp with suitable provision for supporting and releasing it slowly without jerking. The average rate of separation shall not exceed 1 in. per min. The separation shall be noted for a period of 4 min., unless the small size of the specimen makes a shorter period necessary.

(*b*) *Strip Specimens.*—The various parts to be tested shall be separated at one end of the strip specimen a sufficient distance to permit attaching the jaws of the testing clamp. The strip specimen shall be suspended by the separated end, all separate parts of which, except the one under test, shall be clamped in the jaws of the testing clamp. A minimum weight sufficient to maintain the strip specimen in approximately a vertical position shall be attached to the lower end of the test specimen, or the specimen may be held against a vertical plate in such a manner as to hold the specimen in approximately a vertical position during the test. The specified weight shall be applied to the separated end with suitable provision for releasing it slowly without jerking. The average rate of separation shall not exceed 1 in. per min. The separation shall be noted for a period of 4 min.

HYDROSTATIC TESTS

Types of Tests

13. Hydrostatic tests of hose consist of subjecting the specimen to the action of internal water pressure under specified conditions to determine either the bursting strength of the hose or the effect of lower hydrostatic pressures in producing changes similar to those occurring in service. In the latter case, the test may be used as an inspection proof test with a minimum safe limit. Bursting strength is determined on short specimens cut from the full hose lengths but in tests for service effects and for proof inspection, the full lengths are used as specimens. The specimens may be tested, as required, in straight, curved or kinked positions, depending upon the stresses which are desired to be evaluated.

Application of Hydrostatic Pressure

14. Hydrostatic pressure shall be applied by means of a hand or power-driven hydraulic pump or an accumulator system. The hose shall be connected to the water line or pump and filled with water prior to application of pressure, allowing all air in the hose to escape through a petcock provided for the purpose. This is important as a safety measure because expansion of air compressed in the hose, when suddenly released by bursting or other failure, might result in a serious accident. The petcock shall then be closed and the pressure shall be applied at a uniform rate of increase of approximately 1000 lb. per sq. in. per min. Pressure shall be measured with a calibrated gage.

Bursting Test

15. (*a*) The specimen for the hydrostatic test for bursting strength shall be at least 18 in. in length for hose 3 in. and smaller in inside diameter, and at least 24 in. in length for larger hose. The pressure shall be raised as specified in Section 14 until failure, and the pressure at which failure occurs shall be considered the bursting strength of the hose.

(*b*) The curved bursting strength test for cotton rubber-lined fire hose shall be made in accordance with Paragraph (*a*), except that the hose shall be firmly secured at both ends to a suitable test frame which is curved to a radius of 27 in.

Hydrostatic Pressure Tests

16. (*a*) When tests for elongation, contraction, twist, warp rise, and expansion are required, the hose shall be stretched out straight for inspection, and a pressure of 10 lb. per sq. in. applied. All original observations and measurements shall be taken at this pressure. The designated test pressure shall be applied at the rate specified in Section 14 without releasing the original pressure of 10 lb. per sq. in. and final measurements taken. The test pressure shall not be applied longer than 5 sec.

(*b*) *Proof Pressure Tests.*—When proof pressure tests to determine leakage of hose or couplings are required, the proof pressure shall be applied as in Section 14, and held 1 min. The proof pressure shall not exceed 40 per cent of the specified minimum bursting strength.

(*c*) *Kink Tests.*—When kink tests are required, each length of hose selected shall be filled with water with the petcock open to allow all air to escape. The petcock shall then be closed and the hose shall be kinked at one end 18 in. from the coupling by tying the coupling back against the hose so that there will be a sharp kink. The pressure shall then be raised as specified in Section 14 to the required pressure and immediately released.

STEAM TESTS

Types of Test

17. (*a*) Steam tests of hose consist of subjecting test specimens having previously determined physical properties to the action of steam under controlled conditions for known periods after which the physical properties are again measured and the changes noted. In these methods two types of test are given which differ in the manner in which the specimens are exposed to the action of the steam. In one type, named the rack method, the steam is in contact only with the interior of the hose while in the other type, called the digester method, the specimen is entirely surrounded by steam. The choice of type of test to be used for a given hose sample depends on the kind of service being evaluated.

(*b*) Tensile strength and ultimate elongation of the rubber parts and strength of adhesion are the physical properties used in addition to visual and manual inspection for evaluating the effect of the steam. These shall be determined in accordance with Sections 6 to 11, inclusive.

Rack Method

18. (*a*) *Apparatus.*—Two fixed horizontal steam headers having suitable connections for attaching specimens shall be placed one above the other at such distance that the specimens will just fit between the connections in a vertical position without distortion. Dry saturated steam at the required pressure shall be supplied to the specimens through the upper header which shall be equipped with a pressure regulating valve, a recording gage and suitable indicating gages. The lower header shall be connected to a steam trap. Shutoff valves shall be provided at each opening in each header.

(*b*) *Test Specimen.*—The test specimen shall be a piece of the hose sample cut to length as required by the apparatus except that the length shall not be less than 16 in. nor more than 24 in.

(*c*) *Procedure.*—For determining the physical properties before steaming, a portion of the hose sample not required for the rack test shall be used. The specimen for steaming shall be mounted in the apparatus and subjected to internal steam pressure of 75 ± 2 lb. per sq. in. for 7 hr. on each of two successive days, the steam being turned off and the hose allowed to cool during the 17-hr. intermediate interval. After the second steaming period, the hose shall be removed from the apparatus, allowed to cool and held under laboratory atmospheric conditions for not less than 12 hr. nor more than 48 hr. after which the physical properties after steaming shall be determined.

Digester Method

19. (*a*) *Apparatus.*—For the digester test, a suitable pressure vessel having a steam inlet with a regulating valve and a condensate outlet through a steam trap shall be provided. The vessel shall have a petcock air vent for escape of trapped air at the beginning of the test and shall be equipped with a recording thermometer or pressure gage. A perforated metal or wire frame shall be provided for holding the specimens at least 6 in. above the condensate outlet.

(*b*) *Test Specimen.*—The test specimen shall be a piece of the hose sample cut to such length that all test specimens for physical properties can be obtained after discarding at least 2 in. at each end.

(*c*) *Procedure.*—For determining the physical properties before steaming, a portion of the hose sample not required for the digester test shall be used. The specimen for steaming shall be placed in the digester and surrounded by dry saturated steam at a pressure of 45 ± 3 lb. per sq. in. for 48 hr. continuously. At the conclusion of this period, the pressure in the digester shall be released, the hose removed and examination made at once for blistering of the inner tube or cover or loosening of the tube from the fabric. The specimen shall be allowed to cool and to rest under laboratory atmospheric conditions not less than 12 hr. nor more than 48 hr. after which the physical properties after steaming shall be determined.

IMMERSION TESTS FOR SWELL AND DETERIORATION OF HOSE USED FOR PETROLEUM PRODUCTS

Types of Test

20. Immersion tests for evaluating the swelling and deteriorating effects of petroleum products on rubber hose consist of subjecting suitable test specimens from the rubber portion having known physical properties to the action of selected solvents by immersing the specimens completely in solvent under controlled conditions for definite periods after which the physical properties are again determined and the changes noted. In these methods two types of test are given. One type involves measurement of the change in volume of the rubber caused by the swelling action of the solvent. In the other type, the change in strength is measured. For the latter, alternative methods using different solvents are given. In any of the immersion tests, the choice of method and solvent depends upon the nature of the actual service and the petroleum product which is there being used.

Gasoline Immersion Swelling Test

21. (*a*) *Test Specimens.*—The test specimens shall be rectangular rubber blocks 2 by 1 in. having a thickness not over $\frac{1}{16}$ in. They shall be cut from the hose and buffed on both faces only to the extent necessary to insure smoothly buffed faces, except when the material is too thick, in which case the buffing shall be sufficient to reduce the specimens to $\frac{1}{16}$ in. Three specimens shall be used for each test and the results averaged.

(*b*) *Procedure.*—The volume of each test specimen shall be measured by the water displacement method in which the specimen is accurately weighed to the nearest milligram in air (W_1) and in distilled water (W_2) at room temperature. When weighing in water, care must be taken that the specimen is free from adhering air bubbles and, if necessary, it may first be wetted by being dipped in 95 per cent ethyl alcohol and thoroughly rinsed with distilled water. After weighing, the specimen shall be blotted dry with filter paper, completely immersed in gasoline and allowed to stand for 24 hr. at room temperature. The gasoline used shall be straight run aviation grade as described in Section 8 (*a*). Excessive evaporation may be avoided by fitting the container loosely with a cork stopper. At the termination of the immersion period, the specimen shall be removed from the gasoline, dipped in 95 per cent ethyl alcohol, blotted lightly with filter paper and immediately weighed in distilled water (W_3) as before. The final weighing shall be completed within 5 min. after removal from gasoline.

(c) *Calculation.*—The volume increase shall be calculated as follows:

$$\text{Percentage Increase in Volume} = \frac{W_2 - W_3}{W_1 - W_2} \times 100$$

Kerosine-Benzol Immersion Strength Deterioration Test

22. (a) *Test Specimens.*—Six dumbbell-shaped test specimens, prepared as described in Section 8, will be required. Both faces of the dumbbells shall be buffed.

(b) *Apparatus.*—The apparatus in which a specimen is subjected to the action of the solvent shall consist of a glass container fitted tightly with a cork stopper and an efficient reflux condenser. The specimen shall be supported at least ½ in. from the bottom of the tube by means of a wire screen or similar device.

(c) *Kerosine.*—The kerosine shall be of a grade conforming to the following requirements:

TEST	A.S.T.M. DESIGNATION[a]	LIMIT
Burning quality test, hr.	D 187	16 min.
Flash point, deg. Fahr.	D 56	115 min.
End point, deg. Cent.	D 86	330 max.
Cloud point, deg Fahr.	D 97	5 max.
Sulfur, per cent	D 90	0.13 max.
Color, Saybolt number	D 156	+16 min.

[a] These designations refer to the following methods of the American Society for Testing Materials:
Standard Method of Test for Burning Quality of Kerosine Oils (A.S.T.M. Designation: D 187),[1]
Standard Method of Test for Flash Point by Means of the Tag Closed Tester (A.S.T.M. Designation: D 56),[2]
Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86),[3]
Standard Method of Test for Cloud and Pour Points (A.S.T.M. Designation: D 97),[4]
Tentative Method of Test for Sulfur in Petroleum Oils by Lamp Method (A.S.T.M. Designation: D 90 - 34 T),[5] and
Tentative Method of Test for Color of Refined Petroleum Oil by Means of the Saybolt Chromometer (A.S.T.M. Designation: D 156 - 34 T).[5]

(d) *Benzol.*—The benzol shall be a pure, water white grade, free from suspended matter and conforming to the following requirements:

Specific gravity at 60 F.	0.875 to 0.886
Distillation limits:	
Initial boiling point	168 to 172 F.
95 per cent evaporated	174 F., max.
Dry	176 F., max.
Recovery	98 per cent, min.

(e) *Procedure.*—Three specimens shall be tested for tensile strength and elongation in accordance with Section 9 and the results averaged to give the properties of the rubber before immersion. The remaining three specimens shall be placed in the immersion apparatus and completely surrounded by solvent consisting of 85 per cent kerosine and 15 per cent benzol. With the reflux condenser in place the apparatus containing the specimen shall then be maintained at a temperature of 135 to 140 F. (57 to 60 C.) for 48 hr. Use of a thermostatically controlled liquid bath is recommended. At the end of this period, the specimens shall be removed, dipped once in acetone at room temperature and blotted lightly with filter paper. The specimens shall then be suspended or placed on a screen and allowed to dry at room temperature in air protected from drafts for 24 hr. after which the tensile strength and elongation after immersion shall be determined as before, except that the measurements for cross-sectional area and test length between bench marks shall be taken after immersion and drying. The deterioration shown by the difference between the determinations before and after immersion shall be expressed as a percentage of the original property.

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 834.

[2] *Ibid.*, p. 907.

[3] *Ibid.*, p. 870.

[4] *Ibid.*, p. 853.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, pp. 911, 895 (1934); also 1937 Book of A.S.T.M. Tentative Standards, pp. 751, 720.

Oil Immersion Strength Deterioration Test

23. This determination shall be made exactly as in the case of the kerosine-benzol test described in Section 22, except that the temperature during immersion shall be maintained at 200 F. ± 2 F. (93 C. ± 1 C.) for 48 hr. and the solvent shall be a neutral, pale, paraffin-base oil. The oil shall be yellow in color with blue fluorescence, free from turbidity and shall conform to the following requirements:

TEST	A.S.T.M. DESIGNATION[a]	MINIMUM	MAXIMUM
Specific gravity, per cent...............	.....	0.878	0.882
Flash point, deg. Fahr..................	D 92	330	340
Saybolt Universal Viscosity at 122 F., sec.	D 88	61	66

[a] These designations refer to the following methods of the American Society for Testing Materials:
Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).
Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).[2]

INSPECTION AND REJECTION

Inspection

24. (*a*) All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified. The manufacturer shall afford the inspector, without charge, all reasonable facilities to test and inspect the hose.

(*b*) The purchaser may make the tests and inspection to govern the acceptance or rejection of the material in his own laboratory or elsewhere. Such tests and inspection shall be made at the expense of the purchaser not later than 60 days after receipt of the material.

Retests and Rejection

25. (*a*) Any material which fails in one or more tests may be resampled and retested at the expense of the manufacturer. For this purpose two

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 892.
[2] *Ibid.*, p. 982.

additional samples shall be selected from the hose for this test which failed to meet the requirements. Failure of either of the retested samples shall be cause for final rejection.

(*b*) Rejected material shall be disposed of as directed by the manufacturer and at his expense.

TENTATIVE METHODS OF TESTING RUBBER INSULATED WIRE AND CABLE[1]

A.S.T.M. Designation: D 470 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.[2]

Scope

1. These methods are intended for use in testing all types of rubber-insulated wire and cable. They are not to be considered completely applicable to all types of wire and cable, nor do they necessarily include every test applicable to a particular type. To determine the tests to be made on a particular wire or cable, reference should be made to the specifications for that type.

PHYSICAL TESTS OF INSULATION

Scope

2. Physical tests include the determination of tensile strength, tensile stress, ultimate elongation, permanent set, and accelerated aging.

Sampling

3. (*a*) *Number of Samples.*—Wire and cable shall be sampled as follows:

For sizes less than 250,000 cir. mils, one sample shall be selected for each quantity ordered between 2000 ft. and 50,000 ft. of wire or cable and one additional sample for each additional 50,000 ft. thereafter. No sample shall be selected from lots of less than 2000 ft.

For sizes 250,000 cir. mils and over, one sample shall be selected for each quantity ordered between 1000 ft. and 25,000 ft. of wire or cable and one additional sample for each additional 25,000 ft. thereafter. No sample shall be selected from lots of less than 1000 ft.

(*b*) *Size of Samples.*—Samples shall be at least 6 ft. in length where the wire size is less than 250,000 cir. mils, and at least 3 ft. in length where the wire size is 250,000 cir. mils or over.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

[2] Accepted for publication as tentative by Committee E-10 on Standards, August 26, 1937.

Test Specimens

4. (*a*) *Number.*—From each of the samples selected in accordance with Section 3, test specimens shall be prepared as follows:

		NUMBER OF TEST SPECIMENS
For determination of initial properties (unaged)	Tensile strength, tensile stress, and ultimate elongation	3
	Permanent set	3
For aging tests	Air pressure heat or oxygen pressure	3
	Geer oven	3

One specimen of each three shall be tested and the other two held as spares except that where only one sample is selected all three specimens shall be tested and the average of the results reported.

(*b*) *Size.*—In the case of wire and cable smaller than No. 6 A.w.g. with an insulation thickness less than $\frac{3}{32}$ in., the test specimen may be the entire section of the insulation. Where the full cross-section is used, the sample shall not be cut longitudinally. In the case of wire and cable of No. 6 A.w.g. and larger, or in the case of wire and cable smaller than No. 6 A.w.g. having an insulation thickness greater than $\frac{3}{32}$ in., samples approximately square in section, with a cross-section not greater than 0.025 sq. in., shall be cut from the insulation. In extreme cases, it may be necessary to use a segmented or sector-shaped specimen. The test specimens shall be approximately 6 in. in length.

(*c*) *Preparation.*—The test specimen shall have no surface incisions and be as free as possible from other imperfections. Surface irregularities, such as corrugations due to stranding, etc., shall be removed by buffing. When a test specimen is buffed, care shall be taken to make it smooth and of uniform thickness.

NOTE.—The removal of the rubber insulation can be greatly accelerated and in most cases a test specimen which is an entire section can be obtained free from surface incisions and imperfections by means of mercury. The mercury should be introduced at one end of the sample between the insulation and the tinned surface of the conductor, and the sample inclined on a support with the end to which the mercury is applied at the top. The separation of the rubber insulation results from the amalgamation of the tin of the conductor with the mercury. The amalgamation is assisted by first immersing and rubbing the tinning on the exposed end of the conductor in the mercury.

(*d*) *Condition and Age.*—Samples of the insulated wire or cable for physical and accelerated aging tests shall be taken after vulcanization and prior to the application of any covering except that a cable tape may be applied before vulcanizing. No tests shall be made within 24 hr. nor later than 60 days after vulcanization unless agreed to by the manufacturer. Specimens shall not be heated, immersed in water, or subjected to any mechanical or chemical treatment not specifically prescribed in these specifications. Specimens for accelerated aging tests having cable tape applied prior to vulcanization shall be aged with such tape removed.

Calculation of Area

5. Calculation of the area of the test specimens shall be made as follows:

(*a*) Where the total cross-section of the insulation is used, the area shall be taken as the difference between the area of the circle whose diameter is the average outside diameter of the insulation and the area of the conductor. The area of a stranded conductor shall be calculated from its maximum diameter.

(*b*) Where a slice cut from the insulation by a knife held tangent to the wire is used, and the slice so cut has the cross-section of a segment of a circle, the area shall be calculated as that of the segment of a circle whose diameter is that of the insulation. The height of the segment is the wall of insulation on the side from which the slice is taken. (The values may be obtained from a table giving the areas of segments of a unit circle for the ratio of the height of the segment to the diameter of the circle.)

(*c*) Where the cross-section of the slice is not a segment of a circle, the area shall be calculated from a direct measurement of the volume or from the specific gravity and the weight of a known length of the specimen having a uniform cross-section.

(*d*) Where a portion of a sector of a circle has to be taken where the conductor is large and the insulation thin, the area shall be calculated as the thickness times the width. (This applies either to a straight test piece or one stamped out with a die, and assumes that corrugations have been removed by buffing.)

(*e*) Where a portion of a sector of a circle has to be taken where the conductor is large and the insulation thick, the area shall be calculated as the proportional part of the area of the total cross-section.

(*f*) The dimensions of specimens to be aged shall be determined before the aging cycle is begun.

Physical Test Procedures

6. (*a*) Except as otherwise specified in these methods, the physical properties shall be determined in accordance with the Tentative Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412-36 T) of the American Society for Testing Materials.[1]

(*b*) The samples to be tested shall be at a temperature of 20 to 28 C.

(*c*) For all physical tests, specimens shall be marked with gage marks 2 in. apart and placed in the jaws of the testing machine with a maximum distance between jaws of 4 in.

(*d*) Tensile stress shall be determined as that load per unit area which produces the elongation of the sample required by the specifications.

(*e*) Permanent set shall be determined after samples have been held at the required elongation for the specified time.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1105 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1266.

Aging Tests

7. (*a*) Except as otherwise specified in these methods, samples shall be aged in accordance with the Tentative Methods of Test for Accelerated Aging of Vulcanized Rubber (A.S.T.M. Designation: D 428 – 36 T) of the American Society for Testing Materials.[1]

(*b*) The period of aging shall be as stated in the specifications.

(*c*) In not less than 16 hr. nor more than 48 hr. after the completion of the aging process, the aged samples shall be subjected to tensile strength and ultimate elongation tests in accordance with Section 6. Physical tests on both aged and unaged specimens shall be made at the same time.

Retests

8. If any specimen fails to meet the values which may be specified for any test, either before or after aging, that test shall be repeated on two additional specimens from the same sample.

Report

9. The report shall include the following:

(*a*) The calculated values of tensile strength, tensile stress, ultimate elongation, and set,

(*b*) All observed and recorded data on which the calculations are based,

(*c*) The date of vulcanization of the rubber, if known,

(*d*) The dates of all tests,

(*e*) The ambient temperatures during the period of physical testing,

(*f*) The type of testing machine used,

(*g*) The method of aging, and

(*h*) The time of aging.

ELECTRICAL TESTS OF INSULATION

Scope

10. Voltage tests include the following:

A. Voltage Test **on** *Entire Cable*

B. Double-Voltage Test on Short Samples

C. Cold-Bend, Long-Time Voltage Test on Short Samples

Apparatus

11. (*a*) Tests may be made with either alternating or direct voltage.

(*b*) For tests with alternating voltage, there shall be used a transformer of such capacity that the wave form is not appreciably distorted from a sinusoidal shape when loaded with the test specimen. Voltage shall be measured by a tertiary coil and voltmeter or by an electrostatic voltmeter or potential transformer with suitable low voltage indicator, connected to the secondary, or high voltage side, of the test transformer. The frequency of the test voltage shall be nominally between 25 and 60 cycles per second, inclusive.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 1113 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1274.

(*c*) For tests with direct voltage, there shall be used a battery, generator or suitable rectifying equipment supplied with alternating current. Voltage shall be measured with an electrostatic voltmeter, or in the case of the rectifying equipment, with suitable low voltage indicators, provided the latter are so connected that their indications are independent of the test load. The test rectifier shall at all times be so loaded, if necessary by the use of additional capacitive ballast, that the direct current ripple does not exceed 4 per cent.

Application of Voltage

12. (*a*) *Single Conductor Cables.*—Single conductor cables shall be tested between conductor and metallic sheath or water in which they are immersed.

(*b*) *Multiple Conductor Cables.*—In the case of multiple conductor cables, each conductor shall be tested against adjacent conductors and against the sheath or water.

Rate of Application

13. Voltage shall be increased from zero to the prescribed test value at a uniform rate of rise approximating but not exceeding 3 kv. per sec.

A. Voltage Test on Entire Cable

14. Wire and cable shall be subjected to alternating voltages as may be specified or to direct voltages equal to 2.2 times the specified alternating voltages. The duration of test shall be 5 min.

15. Tests shall be made as follows:

(*a*) *General.*—Where insulated conductors are subjected to a high voltage test in water they shall be immersed in a grounded water bath and tested while still immersed.

(*b*) *Single Conductor.*—Single conductors shall be tested after 12-hr. immersion and without covering except a tape or braid applied before vulcanization.

Rubber sheathed conductors shall be immersed and tested after the sheath is applied.

Where a metallic sheath is applied, the conductors shall be retested against the sheath.

(*c*) *Multiple Conductor.*—The individual conductors of multiple conductor cables shall be tested before assembling as prescribed in Paragraph (*b*).

After assembly, each conductor shall be tested against the adjacent conductors. Where a metallic sheath is applied each conductor adjacent to the sheath shall, in addition, be tested against the sheath.

Cables having a rubber or other non-metallic waterproof sheath shall be immersed for 12 hr. and the conductors tested against ground. Cables covered with cable tape and braid or double braid with a weather-resistant finish shall not be immersed for testing.

Cables having a metallic armor shall be tested after the application of the armor at a voltage equal to 80 per cent of the test voltage before armoring. Such cables shall be immersed for 12 hr. prior to test only if for submarine use, and if no lead sheath or shielding is applied.

B. *Double-Voltage Test on Short Samples*

Sampling

16. One sample, 10 ft. or more in length as may be required, shall be taken from each 10,000 ft. or fraction thereof of each type of cable, with a minimum of three samples. Tests upon lots of less than 10,000 ft. shall be made only when specifically requested in the purchase order. Samples may be taken either from process or from completed cable, at the option of the manufacturer.

Procedure

17. At least one-half of the total length of each test specimen shall be in contact with the grounded terminal during the period of test. This part of each specimen shall be immersed in water at room temperature, after the removal of any metallic coverings, for a period of 1 hr. prior to testing. At the end of this time, each test specimen shall be subjected for 5 min. to a voltage equal to twice that specified in Section 14.

C. *Cold-Bend, Long-Time Voltage Test on Short Samples*

Sampling

18. One 10-ft. sample shall be taken from each 10,000 ft. or fraction thereof of completed cable. Tests upon lots of less than 10,000 ft. shall be made only when specifically requested in the purchase order.

Procedure

19. (*a*) Each sample shall be subjected to a temperature of −10 C. for a period of 2 hr. Immediately at the end of this period, the sample shall be bent 180 deg. around a cylindrical mandrel and then straightened. It shall then be bent 180 deg. around the mandrel in the opposite direction. The cable shall be so held during bending operations that it cannot revolve around its own axis.

(*b*) The diameter of the mandrel shall be determined by the following table:

THICKNESS OF CONDUCTOR INSULATION, SIXTY-FOURTHS OF AN INCH	MANDREL DIAMETER AS A MULTIPLE OF OVER-ALL CABLE DIAMETER	
	UP TO 500,000 CIR. MILS	500,000 CIR. MILS AND OVER
10 to 12	8	10
13 to 20	10	12
21 and over	12	12

(*c*) Immediately following the bending test, the cable, while still bent, shall be subjected to the test voltage specified in Section 14 for a period of 2 hr.

Report

20. The report shall include the following:

(*a*) The type of test,
(*b*) The time and place of test,
(*c*) The test voltages,
(*d*) The duration of each test, and
(*e*) The result of each test, including location of any failure.

Insulation Resistance Tests

Apparatus

21. (*a*) The apparatus shall consist of a source of constant potential of from 100 to 500 v., a galvanometer with suitable shunts, and a calibrating resistance of at least 0.1 megohm.

(*b*) The galvanometer should have a high current sensitivity. A sensitivity of the order of 10^{-9} amp. per cm. with scale at a meter distance is desirable. The most convenient shunt is the type known as a universal shunt whereby the current through the galvanometer may be changed by powers of ten without changing its damping.

Insulation Resistance Test Procedure

22. (*a*) Single conductor cables shall be tested between conductors and sheath or water in which they are immersed. Tests on multiple conductor cables with unshielded conductors shall be made between each conductor and all other conductors and the sheath or water. Tests on multiple conductor cables with shielded conductors shall be made between each conductor and shield. The conductor of the wire or cable shall be connected to the negative terminal, and the sheath and other conductors, if any, to the positive terminal of the apparatus. On short sections of wire or cable, a guard circuit should be used to prevent end leakage.

(*b*) The deflection of the galvanometer caused by passing current through the calibrating resistance shall first be determined. The wire or cable shall then be connected into the circuit. At the end of 1 min. after closing the circuit, the galvanometer deflection shall again be noted, and the temperature of the wire or cable observed.

Calculations

23. Insulation resistance is commonly expressed in terms of a standard length. The insulation resistance of a standard length may be determined from the following formula:

$$R = M\frac{D}{d}\frac{S}{s}\frac{l}{l_s}$$

where M = the calibrating resistance,
D = the deflection produced with calibrating resistance in circuit,
S = the shunt ratio with calibrating resistance in circuit,
d = the deflection produced with test specimen in circuit,
s = the shunt ratio with test specimen in circuit,
l = the length of entire wire or cable, and
l_s = the standard length.

Temperature Correction

24. The insulation resistance of wires and cables varies widely with temperature. If the temperature at which the measurement was made differs from 60 F. (15.6 C.), the resistance shall be reduced to that at 60 F. (15.6 C.) by multiplying the measured value by the temperature coefficient specified for the insulation in question.

Moisture Absorption Test

Moisture Absorption Test Procedure

25. (*a*) A 15-ft. sample of the insulated wire or cable shall be taken after vulcanization and prior to the application of any coverings except tape applied before vulcanizing. Such cable tape shall be removed before making the moisture absorption test. The middle 10 ft. of the sample shall be immersed in distilled water for a period of 14 days with the $2\frac{1}{2}$-ft. portion at each end kept above the water as leakage insulation. The specific inductive capacity of the insulation shall be taken after 1, 7, and 14 days with the water at the same temperature for each measurement. The water shall be maintained at room temperature but not less than 70 F. (21 C.).

(*b*) *Calculations.*—The specific inductive capacity shall be determined at commercial frequencies and shall be calculated as follows:

$$\text{Specific inductive capacity} = 13{,}600\ C \log_{10}\frac{D}{d}$$

where C = the capacity in microfarads of 10 ft. of sample,
D = the diameter over the insulation, and
d = the diameter over the conductor.

The moisture absorption test shall be made only when requested by the purchaser.

TENTATIVE METHODS FOR THE IDENTIFICATION OF FIBERS IN TEXTILES AND FOR THE QUANTITATIVE ANALYSIS OF TEXTILES[1]

A.S.T.M. Designation: D 276 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1927; REVISED, 1929, 1930, 1931, 1933, 1935, 1937.

METHODS FOR THE IDENTIFICATION OF FIBERS IN TEXTILES[2]

Successful identification of the fibers used in textiles depends upon experience and familiarity with the fibers. The ultimate test of the diagnosis of an unknown is comparison with a known standard sample. It is not only desirable but necessary for the analyst to have authentic samples of the fibers likely to occur in the textiles he is called upon to analyze.

The key used in these methods is not concerned with describing the fiber but rather with pointing out for each group of fibers and finally, within the group, for each fiber, that particular characteristic or group of characteristics by which the fiber differs from similar fibers. Often this is a minor detail of structure, or staining reaction. Occasionally, it is somewhat subjective, but an effort has been made to choose objective characteristics which will be evident to all observers. For the benefit of those unfamiliar with the use of a key it should be said that the arrangement is necessarily artificial. Groups are established on the basis of similarity and dissimilarity for convenience only, and without regard for chemical, biological or industrial relationships.

The "keying out" of an unknown is simply a matter of elimination. In every division and subdivision of the key there are *two alternatives* both numbered or lettered alike. It is necessary to *reject* one and to *accept* the other as descriptive of the fiber in question. It is quite possible that neither phrase exactly describes the fiber, but it is necessary to make the best choice between the two and to follow through the entire key, step by step, in the same manner. A decision should never be made as to which group the fiber belongs until both descriptions under that designation have been considered. Except where definitely stated, descriptions are of ultimate fibers rather than of "technical" fibers or fiber aggregates. For adequate study of these a microscope is necessary, and for analytical purposes a magnification of 100 diameters is most satisfactory.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

[2] Attention is called to the Photomicrographs of Common Textile Fibers appearing in Appendix I, of the volume, "A.S.T.M. Standards on Textile Materials," p. 221; available as a separate publication, September, 1937.

The technique of mounting fibers for microscopical examination is relatively simple, but experience brings proficiency and speed. To remove dyes, natural or applied oils, starch, sizing, and other materials it is advisable to begin by heating the fibers in a dilute solution of caustic soda (0.5 per cent). Because wool and hair are quickly disintegrated in hot caustic, care shall be taken to shorten the cooking in such cases. In examining fabrics this step is often not necessary. In vegetable fibers prolonged boiling is beneficial in breaking up the fiber bundles and larger aggregates so that the ultimate cells can be separated. Since most stains are indicators, it is necessary that all caustic be washed from the sample before proceeding further with the tests. Rubbing and squeezing between the fingers under running tap water is sufficient. Small samples of the fiber shall be placed on a slide and teased apart with needles. Too much material on one slide renders adequate separation difficult. It cannot be too strongly emphasized that a just and impartial consideration of the material must be based on single fibers and their minute configurations rather than on a confused mass of fibers. Moreover, several small samples taken at random from various regions of the material give more representative results than a single sample. Most stains take better if excess moisture has been blotted from the fiber with filter paper. To be sure that all fibers are stained equally they shall be stirred on the slide with needles. The most satisfactory examination of stained fibers is by transmitted light. Constant adjustment of the focus discloses the lumen and other structural features which may otherwise escape notice; and decreasing the intensity of the light by means of diaphragm or condenser adjustment is sometimes a revealing manipulation. It is worth while to notice extraneous materials which occur with the fiber. The presence or absence of epidermal cells, spirals, hairs, unusual configurations in cell groups or fiber bundles is often a clue when other features are less distinct than usual. An experienced fiber analyst has always in mind certain associations which though of no technical value, are to him, personally, dependable means of identification.

REAGENTS

The following reagents will be required:

Aniline Sulfate

Aniline sulfate	1 g.
Distilled water	100 ml.
Sulfuric acid (10 per cent solution)	1 ml.

Hemp stains yellow; linen is unstained.

Diphenylamine Solution

Concentrated H_2SO_4	20 ml.
Glacial acetic acid	10 ml.
Diphenylamine	0.3 g.

Hahn's Solution

Solution A:

Picric acid	1 g.
Distilled water	100 ml.

Solution B:

Soluble Blue 2B Extra (Color index 707)	**0.2 g.**
Eosin (Color index 768)	**0.1 g.**
Tannic acid	**1.0 g.**
Distilled water	**100 ml.**
Hydrochloric acid (10 per cent solution)	**0.2 ml. (when solution has cooled)**

Mix solutions *A* and *B* in equal parts for rayon tests.

Herzberg's Solution

Solution A:

Zinc chloride	50 g.
Distilled water	25 ml.

Specific gravity at 28 C. should be 1.8

Solution B:

Potassium iodide	5.25 g.
Iodine	0.25 g.
Distilled water	12.5 ml.

Add solution *B* to solution *A*, stir; let stand overnight; decant from sediment; add a leaf of iodine; keep in dark bottle.

A good general stain for all fibers. Linen, cotton, hemp, ramie are stained red; jute, phormium, sisal and other lignified fibers, yellow. De-lignified fibers and cellulose cells are stained blue.

Herzog's Cyanin

Saturated alcoholic solution of Cyanin Blue (Color Index 288)	80 ml.
Distilled water	10 ml.
Concentrated glycerine	30 ml.

Apply hot to fibers; mount fibers in concentrated glycerine. Flax fibers are unstained; hemp fibers are blue.

Millon's Reagent

Dissolve mercury in an equal weight of cold fuming HNO_3 (sp. gr. 1.4), then apply moderate heat.

Dilute solution with 2 volumes of water.

Picric Acid

Picric acid	**1 g.**
Distilled water	**100 ml.**
Sodium hydroxide	**0.5 g.**

Brings out surface scales on animal fibers, especially wool and mohair.

ABRIDGED KEY FOR COMMON FIBERS

The fibers commonly met in textile fabrics are cotton, linen, jute, rayon, silk and wool. A few simple tests will identify these, but it is advisable to confirm the diagnosis by other tests rather than to rely on one identification only.

A. As a preliminary test, burn a few individual fibers by applying a flame to their ends.

Odor of burned feathers or hair; knob forms on end of fiber.......... *Silk or Wool* (see *a*).
Odor of burned feathers or hair; ash retains shape of thread.......... *Weighted Silk* (see *a*).
Distinctly aromatic odor, somewhat acrid; knob forms on end of thread.......... *Acetate Rayon* (see *b* and *c*).
Odor of burned paper or straw; ash shapeless; drops off.......... *Other Rayons, Cotton, Linen or Jute.*

B. Immerse fibers in an aqueous solution of caustic soda (0.5 per cent) and bring the solution to a boil. Wash thoroughly by squeezing and rubbing between the fingers under running tap water. Tease the fibers apart on a slide, blot off water with filter paper. Stain with a few drops of Herzberg's solution. Examine under magnification of 100 diameters.

1 Fibers yellow, white or greenish.......... 2.
  2. Fibers distinctly pointed at ends, with narrow lumen; bright golden yellow.......... *Jute.*
  2. Fibers of indeterminate length, with cut ends.......... 3.
    3. Fibers fine, sometimes flattish, of varying diameters, transparent, without cellular structure.......... 4.
      4. Fibers with longitudinal striations, and sometimes with paired parallel lines in a diagonal direction (imprint of one fiber across another).......... *Wild* (*Tussah*) *Silk* (see *a*).
      4. Fibers with fine diameters, without markings.......... *Cultivated Silk* (see *a*).
    3. Fibers larger, rod-shaped, with prominent scales.......... *Wool* (see *a* and *d*).
1. Fibers reddish or purple.......... 5.
  5. Fibers cylindrical, pointed at ends, with cross marks on surface; swollen nodes, and narrow lumen.......... *Linen* (see *e*).
  5. Fibers lacking above characteristics.......... 6.
    6. Fibers flat, more or less ribbon-like, frequently twisted, with broad lumen.......... *Cotton* (see *e*).
    6. Fibers without any lumen, straight.......... *Rayon* 7.
      7. Fibers with distinct longitudinal striations on the surface.......... *Viscose.*
      7. Fibers without striations.......... 8.
        8. Stain with Hahn's solution. Fibers pink.......... *Nitrocellulose Rayon* (see *f*).
        8. Fibers not pink.......... 9.
          9. Fibers yellow.......... *Cellulose Acetate Rayon* (see *b* and *c*).
          9. Fibers blue or colorless.......... *Cuprammonium Rayon* (see *g*).

(*a*) Millon's reagent colors true silk fibers (or other animal fibers such as wool, mohair or fur) pink. Vegetable fibers are not stained.

(*b*) Cellulose acetate rayon is dissolved in acetone; other fibers are not affected.

(*c*) Cellulose acetate rayon is dyed yellow in picric acid; other rayons wash out white.

(*d*) Surface markings, scales, etc., on wool fibers become more pronounced when fibers are stained with 1 per cent picric acid containing sodium hydroxide.

(*e*) Untwist the thread until a single dry fiber is exposed. Moisten the tip between thumb and finger; observe with a hand lens, as follows:

Fiber tip curls clockwise........................linen.
Fiber tip curls counter-clockwise................cotton.

(*f*) Nitrocellulose rayon dissolves to give a deep blue coloration when tested dry with a solution of diphenylamine in sulfuric acid.

(*g*) Cuprammonium rayon is dyed deep orange in 0.2 per cent solution of Erie Fast Orange C. G. (Color index 621); other rayons are white or weak orange.

ANALYTICAL KEY FOR IDENTIFICATION OF FIBERS

Touch end of fiber to a flame:

Fiber does not burn..........................Mineral Origin I.
Fiber burns with characteristic odor of burning feathers or glue..............................Animal Origin II.
Fiber burns with characteristic odor of burning paper or straw............................Vegetable Origin III.

I. Fibers of Mineral Origin:

Asbestos, confirm by microscopical examination.

II. Fibers of Animal Origin:

1. Fibers coarse, more or less shiny, wiry; usually found in brushes or upholstery..............................2.
2. Fibers with diameters tapering from base to tip.......*Bristles* 3.
3. Bristles dark, with pronounced white band short distance back of tip; tips not flagged..........*Badger Bristle.*
3. Bristles same color throughout, with a knob frequently visible at butt ends where hair was pulled from follicle; tips flagged..............*Hog Bristle* 4.
4. Bristles hardly more than 1½ in. in length; varicolored; frequently curved; irregular as to length and diameter.....................*Domestic Hog Bristle.*
4. Bristles longer than 1½ in., of rather uniform length, *straight*..........................5.
5. Bristles all white, or chalk white............*Bleached "(French)" Bristle.*
5. Bristles black, yellow, or grey (black and yellow mixture).......................6.
6. Bristles 4 in. or longer, stiff, coarse, much flagged, usually grey or yellow........*Russian Bristle.*
6. Bristles all black, 4 in. or shorter..........*Chinese* 7.

7. Bristles black, rather soft, often with flagged end clipped off............. *Chungking Bristle.*

7. Bristles with flagged ends never clipped. 8.

8. Bristles very fine, soft, usually not over 3 in...................... *Tientsin Bristle.*

8. Bristle not so fine, longer............ *Hankow Bristle.*

2. Fibers with rather regular diameters throughout; little or no taper; tips not flagged; hair follicle knobs seldom present.. *Hair* 9.

9. Hairs wiry, all black.............................. 10.

10. Hairs of average diameter of not less than 0.0055 in. (based on microscopical measurement of at least 100 hairs)........................... *Horsetail Hair.*

10. Hairs of average diameter less than 0.0055 in...... *Horse Mane Hair.*

9. Hairs usually white, yellow or brown................ 11.

11. Hairs smooth, scales scarcely visible.......... 12.

12. Hairs, smooth, roundish, showing under magnification of 100, distinct pigmentation, not isolated in a medulla, but scattered in lumpy streaks........... *Horsehair* (see 10).

12. Hairs less smooth, exhibiting less "character" than the above; showing under magnification of 100, distinct medulla with pigment closely packed in flat, accordion-like masses................ *Cattle Hair.*

11. Hairs coarse with prominent, surface scales, wide medulla filled with pigment but not packed in any characteristic arrangement........ *Goat Hair.*

1. Fibers fine, soft, more or less silky; from soft brushes, or from felts and fabrics............................ 13.

13. Fibers fur-like............................ 14.

14. Fibers showing pigmentation in wide medulla with regularly spaced blocks of pigment (×100).................. 15.

15. Hairs rather short, with pigment blocks in 1 or 2 rows.................. *Common Rabbit.*

15. Hairs abnormally long, showing more than 2 rows of pigment blocks.... *Angora Rabbit.*

14. Fibers showing pigment tightly packed in wide medulla, in irregularly checked mass............................... *Squirrel.*

13. Fibers filamentous 16.

16. Fibers rod-shaped with medullary canals, and scaly surfaces (×100)... 17.

17. Fibers tan, brown or black........ 18.

18. Fibers with distinctly granular pigmentation and indistinct hollow medulla...... *Alpaca.*

18. Fibers with no distinct medulla except in kemps where medulla is large and heavily pigmented............ *Camel Hair.*

17. Fibers usually white........................19.
    19. Fibers with fine scales closely appressed to fiber surface; diameter usually small..*Mohair.*[1]
    19. Fibers of various diameters, scales coarser, more loosely arranged...............*Wool.*
16. Fibers flat, smooth, transparent, void of cellular structure..................................20.
    20. Fibers broad, varying in diameter, marked with longitudinal striations, and frequently with parallel diagonal lines where one fiber crossed over another has left an impression.....................*Wild (Tussah) Silk.*
    20. Fibers single, narrow, without striations............................*Cultivated Silk.*

III. Fibers of Vegetable Origin:

For convenience these are grouped according to their uses as. *A. Brush Fibers*, *B. Cordage Fibers*, and *C. Vegetable Fibers from Fabrics.*

A. Brush Fibers[2]

1. Fibers hair-like, or rope-like, white, yellow (or dyed).....*Tampico.*[3]
1. Fibers more woody than hair-like......................2.
    2. Fibers dark brown or black.........................3.
        3. Fibers thick, more or less circular, corky in texture..*Rattan.*
        3. Fibers not corky, but woody.....................4.
            4. Fibers thin, wiry, circular...................*Bassine.*
            4. Fibers thicker, flatter, more woody, brittle.......*Bass.*
    2. Fibers reddish, wiry..............................*Palmetto.*

B. Cordage Fibers

Apply Swett's test as follows:

Wash sample in alcohol to remove grease. Immerse in nascent chlorine solution for 30 sec. Wash in tap water; rinse in alcohol to remove water. Hold over concentrated ammonium hydroxide. Fumes produce:

Cherry red color ....Sisal and other rope fibers.
Brown color...............................Manila fibers.

1. Fibers brown or yellow...............................*Abaca* (Manila).[4]

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[1] Scales may be brought out distinctly by staining with picric acid.

[2] The term "fiber" is here used in its technical sense, meaning not the ultimate fiber cell, but rather a bundle or an aggregate of fibers.

[3] Sisal, palma, istle, henequen and other agaves.

[4] Confirm by microscopical examination for broad lumen.

1. Fibers cherry red or pink..............................2.
    2. Treat fibers in boiling dilute caustic, wash, separate into ultimate cells; stain with Herzberg's solution, examine under magnification of 100. Fibers yellow..............................3.
        3. Fibers more or less rounded at ends, diameters regular, fiber surface strikingly smooth, canal not visible; fiber bundles usually accompanied by spirals..............................(*Phormium Tenax*).
        3. Fibers distinctly pointed at ends, with irregular diameters, thick cell wall, and distinct narrow lumen..............................4.
            4. Fiber bundles accompanied by spirals and epidermal cells..............................*Agave* (Sisal, Henequen, etc.).
            4. Fiber bundles free from spirals. Characteristic surface markings on fiber.........*Jute*.
    2. Fibers red, purple or blue..............................5.
        5. Fibers ribbon-like, flat, twisted, with broad lumen..............................*Cotton*.
        5. Fibers not twisted; lumen narrow; bumps, swellings, cross striations on fiber surface. Red fibers frequently accompanied by yellow epidermal cells (shive)............6.
            6. Stain with Herzog's cyanin (or with Aniline sulfate). Fibers stained.............7.
                7. Stain with Herzberg's solution: Yellow epidermal cells with prominent hook hairs near stomatal openings.......*Sunn Hemp*.
                7. Yellow epidermal cells devoid of hairs...*True Hemp*.
            6. Stained with Herzog's cyanin. Fibers colorless..............................*Flax*.

C. Vegetable Fibers from Fabrics

(Or used in Battings, Cushion Filling, etc.).

1. Fibers silky in appearance, with shiny surface...........2.
    2. Fibers short, very fine, downy; examined under microscope showing broad lumen, air filled; very thin cell wall..............................*Kapok*.
    2. Fibers of indeterminate length; threadlike............3.
        3. Dip for 3 minutes in Hahn's stain, wash in cold water. Fibers pink..............................4.
            4. Fibers deep pink..............................*Nitrocellulose Rayon.*[1]
            4. Fibers pale pink..............................*Viscose Rayon.*[2]
        3. Fibers not pink..............................5.
            5. Fibers yellow..............................*Acetate Rayon.*[3]
            5. Fibers blue..............................*Cuprammonium Rayon.*

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[1] Confirm by test with diphenylamine—sulfuric acid for blue color.

[2] Confirm by microscopical examination for striated surfaces.

[3] Confirm by dissolving in acetone.

1. Fibers not silky.....................................6.
6. Stain with Herzberg's solution, examine microscopically. Fibers yellow...............*Jute.*
6. Fibers red or purple.........................7.
7. Fibers with narrow lumen, swollen nodes, distinct cross markings...............8.
8. Moisten tip of single fiber between thumb and finger:
Fiber curls clockwise..................*Linen.*
8. Fiber curls counter-clockwise...........*Hemp.*
7. Fibers with broad lumen.................9.
9. Fibers broad, flat, with unusually wide diameter; joints and transverse fissures prominent..............*Ramie.*
9. Fibers not broad and flat; diameters more or less uniform............*Cotton* 10.
10. Fibers ribbon-like, twisted.......*Raw Cotton.*
10. Fibers swollen, round, with partially destroyed lumen......*Mercerized Cotton.*

METHODS FOR THE QUANTITATIVE ANALYSIS OF TEXTILES

Scope

1. These methods provide for the quantitative determination of the water, total non-fibrous materials, cellulose acetate rayon, silk, regenerated cellulose rayon, cotton, and wool present in textiles composed of two or more of these materials. Any section of the methods which is not applicable in a particular analysis may be omitted. It is assumed that the analyst knows from qualitative tests what materials are present. All results shall be based upon the weight of the dried material.

Water or Regain

2. (*a*) Unless otherwise specified, samples to be analyzed for water shall be conditioned by exposure to an atmosphere having a relative humidity of 63 to 67 per cent and a temperature of 70 to 80 F. (approximately 21 to 26 C.) until equilibrium is established. The material shall be considered to be in equilibrium when it shows no progressive change in weight.

(*b*) The special equipment for drying specimens to constant weight which is generally available in textile laboratories may be used. The procedure outlined in Paragraphs (*c*) to (*f*) is provided for laboratories not so equipped and shall be used in the settlement of disputes unless some other method is agreed upon by those concerned.

(*c*) A glass weighing bottle of approximately 100-ml. capacity fitted with a ground-glass cover shall be dried at 220 to 230 F. (105 to 110 C.) to constant weight. A satisfactory procedure is to place the bottle and cover separately in the oven. After drying for 1 hr. they shall be transferred to a desiccator and allowed to cool to room temperature. The cover shall then be placed on the bottle which shall then be weighed. The heating, cooling and weighing shall be repeated until the weight is constant to within ± 0.003 g. This is the "weight of weighing bottle." The weighing bottle shall be kept in a desiccator when not in use.

(*d*) A specimen of approximately 5 g. of the material to be tested shall be placed in the weighing bottle which shall then be covered and weighed. By subtracting the "weight of weighing bottle" (Paragraph (*c*)) from this weight, the weight of specimen, A, is obtained.

(*e*) The specimen shall be removed from the weighing bottle, placed on a watch glass in the oven (temperature 220 to 230 F. (105 to 110 C.) as before), and dried for 1.5 hr., then quickly transferred to the weighing bottle, which shall be placed uncovered in a desiccator. When the specimen and bottle have cooled to room temperature, the cover shall be replaced on the bottle which shall then be weighed. The specimen shall be returned to the oven and the drying, cooling and weighing shall be repeated until the weight is constant to within ±0.003 g. By subtracting the "weight of weighing bottle" (Paragraph (*c*)) from this weight, the weight of dry specimen, B, is obtained.

(*f*) *Calculation.*—The amount of water present is usually expressed either as moisture content, percentage of the original weight of the specimen, Eq. 1, or as a moisture regain, percentage of the weight of the dry specimen, Eq. 2. When analysis is made on the dry basis, as in this procedure, the amount of water shall be reported as moisture regain as calculated from Eq. 2:

$$\text{Moisture content, per cent} = \frac{A - B}{A} \times 100 \quad \text{(1)}$$

$$\text{Moisture regain, per cent} = \frac{A - B}{B} \times 100 \quad \text{(2)}$$

where A = the original weight of specimen, and
B = the weight of dry specimen.

Total Sizing, Finishing and Other Non-Fibrous Materials

3. (*a*) The purpose of this procedure is to remove all non-fibrous natural constituents of the fiber and substances added by the manufacturer. Starch, China clay, soaps, some waxes, some non-drying oils and the usual natural constituents are in this category and are

removed by the procedure given. However, general directions for the removal of all possible substances which may be present cannot be given. The delustered rayons and some of the newer finishes present special problems. The analyst will have to meet special cases as they arise. When it is necessary to modify the procedure in order to completely remove non-fibrous constituents, the analyst shall make sure that purified samples of the fibers under consideration do not lose weight when subjected to the same treatment. Ordinarily, samples shall be analyzed in accordance with Paragraphs (*b*) to (*d*).

(*b*) A specimen of approximately 5 g. shall be taken for test. Particular care shall be taken to prevent the loss of fibers in the treatments. The specimen shall be dried at 220 to 230 F. (105 to 110 C.) to constant weight (Section 2 (*e*)). This gives the weight of dry specimen, *B*.

(*c*) The specimen shall then be extracted for 2 hr. with carbon tetrachloride in a Soxhlet or similar extractor. The specimen shall be allowed to dry in the air. It shall then be washed by repeated immersion in hot distilled water, squeezing between each immersion.

(*d*) The specimen shall then be immersed in a 3 to 5 per cent aqueous solution of a starch and protein-solubilizing enzyme preparation at 122 F. (50 C.). It shall be squeezed while immersed, removed and squeezed again, and this procedure repeated at least three times. The purpose is to insure thorough wetting of the fibers by the enzyme solution. The enzyme solution shall then be maintained at 122 to 140 F. (50 to 60 C.), or the optimum temperature range for the particular enzyme used, and the specimen kept immersed in it for 1 hr. A shorter time is permissible if sizing is removed in the shorter time. Fifteen minutes is sufficient for some textiles. The specimen shall then be rinsed twelve times in fresh portions of hot distilled water, squeezing after each rinse. Thorough rinsing is necessary in order to remove china clay from heavily filled fabrics. The enzyme treatment and rinsing may be carried out in a Launder-Ometer or similar device if desired. The rinsed specimen shall then be dried at 220 to 230 F. (105 to 110 C.) to constant weight. This gives the weight of dry fiber, weight *C*.

(*e*) *Calculation.*—The total amount of non-fibrous constituents present shall be calculated from the following formula:

$$\text{Total sizing, finishing and other non-fibrous materials (dry basis), per cent} = \frac{B - C}{B} \times 100$$

where B = the weight of dry specimen (Paragraph (*b*)); and
C = the weight of dry fiber (Paragraph (*d*)).

Cellulose Acetate Rayon

(Acetone Soluble Types Only)

4. (*a*) The specimen after being tested in accordance with Section 3 shall be agitated vigorously for 15 minutes in about 50 times its weight of acetone at room temperature.[1] The residue shall be rinsed while squeezing by alternate immersion in acetone, two fresh portions of acetone being used. The residue shall be allowed to dry and them immersed in water at a temperature of about 158 F. (70 C.). The excess water shall be removed by squeezing and the residue dried to constant weight as described in Section 2 (*e*). This weight is called *D*.

(*b*) *Calculation.*—The cellulose acetate rayon content shall be calculated from the following formula:

$$\text{Cellulose acetate rayon (dry basis), per cent} = \frac{C - D}{B} \times 100$$

where B = the weight of dry specimen (Section 3 (*b*));
C = the weight of dry fiber (Section 3 (*d*)); and
D = the weight of dry specimen after removal of cellulose acetate rayon.

Silk

5. (*a*) The fibers constituting the residue from the test for cellulose-acetate rayon (Section 4) shall be cut to lengths of about 2 to 4 mm. They shall be agitated vigorously for 1 hr. in 200 ml. of a clear aqueous solution of calcium thiocyanate of specific gravity 1.20 to 1.21 at a temperature of 158 F. (70 C.) made just acid to litmus with acetic acid, and maintained at a temperature of 158 ± 4 F. (70 ± 2.2 C.). Precautions shall be taken to prevent evaporation from the solution with consequent concentration of the thiocyanate. The fibers which are not dissolved shall be collected in a small Büchner funnel, Gooch crucible, or bitumen filter, preferably with the aid of suction. When a good pad of fibers has formed on the filter, the hot mother liquor shall be poured through a second time to recover all fibers on the pad. The fibers shall then be agitated for 5 min. in a fresh 200-ml. portion of the thiocyanate solution. The filtration shall be repeated and the fibers washed with hot distilled water until free from thiocyanate. They shall be dried to constant weight, weight *E*.

(*b*) *Calculation.*—The silk content shall be calculated from the following formula:

$$\text{Silk (dry basis), per cent} = \frac{D - E}{B} \times 100$$

where B = the weight of dry specimen (Section 3 (*b*));
D = the weight of dry specimen (Section 4 (*a*)); and
E = the weight of dry specimen after removal of silk.

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[1] A Launder-Ometer can be used to obtain satisfactory agitation.

Regenerated Cellulose Rayon

6. (*a*) The fibrous residue from the test for silk (Section 5) shall be pulled apart or cut up. It shall be treated in accordance with the procedure in Section 5 except that the acid solution of calcium thiocyanate shall be of specific gravity 1.35 to 1.36 at a temperature of 158 F. (70 C.). The weight of the dried residue from this treatment is weight *F*.

(*b*) *Calculation.*—The regenerated cellulose rayon content shall be calculated from the following formula:

$$\text{Regenerated cellulose rayon (dry basis), per cent} = \frac{E - F}{B} \times 100$$

where B = the weight of dry specimen (Section 3 (*b*));
E = the weight of dry specimen after removal of silk (Section 5 (*a*));
F = the weight of the dried residue.

Cotton

7. (*a*) The residue from the test for regenerated cellulose rayon (Section 6), which contains cotton and wool, shall be immersed for 10 minutes in a boiling solution of aluminum chloride containing 5 g. of $AlCl_3$ (or 9 g. of the hydrated salt $AlCl_3 \cdot 6H_2O$) per 100 ml. of water. It shall be removed from the solution and after allowing the excess liquid to drain off (the specimen shall not be squeezed), it shall be heated in an oven at 221 to 230 F. (105 to 110 C.) until the cotton has become brown in color and brittle (usually 2 hr. is sufficient). The specimen shall be placed on a 100-mesh screen and rubbed against the screen with sufficient pressure to powder the carbonized cellulose and pass it through. The residual fibers (wool) shall be placed in a suitable container, preferably a 250-ml. beaker. The material which passed through the screen shall be screened again to recover any wool fibers which passed through. The wool shall be agitated with about 100 ml. of dilute hydrochloric acid solution (1 part by volume of the concentrated laboratory reagent diluted with 9 parts of distilled water), collected as a pad (see Section 5 (*a*)), washed with distilled water until free from chlorides, and dried at 221 to 230 F. (105 to 110 C.) to constant weight, weight *G*.

(*b*) *Calculation.*—The cotton content shall be calculated from the following formula:

$$\text{Cotton (dry basis), per cent} = \frac{F - G}{B} \times 100$$

where B = the weight of dry specimen (Section 3 (*b*));
F = the weight of dried residue (Section 6 (*a*)); and
G = the weight of dry specimen after removal of cotton.

Wool

8. The wool content of the specimen shall be calculated from the following formula:

$$\text{Wool (dry basis), per cent} = \frac{G}{B} \times 100$$

where B = the weight of dry specimen (Section 3 (*b*)); and
G = the weight of dry specimen after removal of cotton.

Wool, Sulfuric Acid Method[1]

9. (*a*) The residue from the test for regenerated cellulose rayon (Section 6), which contains cotton and wool, shall be immersed in 200 ml. of a boiling sulfuric acid solution (which has been carefully prepared to contain 1 per cent of H_2SO_4 by weight) for 7 to 10 min. It shall then be transferred to a crucible having a medium fritted glass bottom and the excess acid solution shall be removed by suction. (A Gooch crucible, without asbestos, may be used to advantage for the filtrations if the liquors passing through at first are returned to the crucible so that all fibers are collected in the pad which forms in the crucible.) The specimen shall be immersed in 200 ml. of a solution of sulfuric acid (which has been carefully prepared to contain 70 per cent of H_2SO_4 by weight) at a temperature of 38 C. and worked in this solution for 15 min. The undissolved fibers shall be collected in the crucible and thoroughly washed with cold water, immersed in a solution of sodium bicarbonate (2 per cent) at room temperature for 5 min. to neutralize any acid, collected again in the crucible, washed, and dried at 105 to 110 C. to constant weight. The weight of the dried residue from this treatment is weight H.

(*b*) If it is not necessary to subject the specimen to the procedures given in Sections 3 to 6, inclusive, the specimen shall be prepared and treated in accordance with Paragraph (*a*) with the following exceptions: Tightly woven, knit, or felt material shall be cut into strips $\frac{1}{8}$ to $\frac{1}{4}$ in. in width. Highly twisted yarns shall be partially untwisted. A specimen weighing approximately 2 g. shall be taken for the test. It shall be dried to constant weight at 105 to 110 C. in accordance with Section 3 (*b*). The weight of the dried specimen is weight B.

(*c*) The wool content of the specimen shall be calculated from the following formula:

$$\text{Wool (dry basis), per cent} = \frac{H}{B} \times 100$$

where B = the weight of the dry specimen (Section 3 (*b*)), and
H = the weight of the dry specimen after removal of cotton.

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[1] This method conforms in substance with the method developed by the American Association of Textile Chemists and Colorists.

Cotton

10. The cotton content of the specimen shall be calculated from either Eq. (1), if the specimen has been treated in accordance with Sections 3 to 6 inclusive, or Eq. (2), if the specimen has been analyzed only for wool and cotton, as follows:

$$\text{Cotton (dry basis), per cent} = \frac{F - H}{B} \times 100 \qquad (1)$$

$$\text{Cotton (dry basis), per cent} = \frac{B - H}{B} \times 100 \qquad (2)$$

where B = the weight of the dry specimen (Section 3 (*b*)),
F = the weight of the dry residue (Section 6 (*a*)), and
H = the weight of the dry specimen after removal of cotton.

TENTATIVE GENERAL METHODS OF TEST
FOR
PROPERTIES OF COTTON FIBERS[1]

A.S.T.M. Designation: D 414 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1935; Revised, 1937.

Scope

1. These methods cover the test procedures for the determination of length, length distribution, fineness, immaturity count, and strength of cotton fibers.

Atmospheric Condition

2. When it is specified that the operations shall be carried out in a "standard atmosphere," an atmosphere having 65 per cent ± 3 per cent relative humidity at 70 F. ± 5 F. (21 C. ± 2.5 C.) shall be considered satisfactory.

Length and Length Distribution

Preparation of Sample

3. (*a*) For making each cotton fiber length array, a composite sample shall be prepared by taking 32 small tufts or pinches from the sample of ginned lint (usually 0.5 to 3.0 lb.) (Note), 16 tufts well distributed from each of the two sides.

Note.—The method of selecting these samples from the gin box, from the various types of bales, or from mill products will depend on the purpose of the study and the quantity of the supply of cotton.

(*b*) The two series of tufts shall be arranged in corresponding rows. Each pair of tufts shall be combined, drawn, lapped in the fingers, and divided by splitting gently into two approximately equal parts, one of which shall be discarded. The resulting tufts shall be arranged in two series in corresponding rows.

(*c*) The process described in Paragraph (*b*) shall be repeated with each successive series containing the 8, 4 and 2 tufts. The two resulting tufts shall be combined, drawn and lapped in the fingers to form a composite tuft.

(*d*) The resulting composite tuft shall be gently cleaned of trash, neps, leaf, etc., and the fibers paralleled and straightened by drawing and lapping in the fingers. Care shall be taken to retain all the fibers except those of neps and tangled masses. In case neps and tangled masses constitute an appreciable proportion of the tuft the percentage by weight shall be determined and recorded.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

(*e*) The sample for sorting shall be obtained by separating from one side of this composite tuft a portion sufficient to weigh approximately 75 mg.

Atmospheric Condition

4. The processes of sorting, measuring and weighing shall be performed in a standard atmosphere (Section 2). (Fibers sorted at relative humidities over or under 65 per cent will measure longer or shorter, respectively, than those sorted at 65 per cent relative humidity.)

Sorting, First Transfer

5. (*a*) The 75-mg. sample shall be carefully inserted into one field of combs (Note) so that it extends across the field at approximately right angles, and occupies a width slightly less than that of the fiber forceps.

NOTE.—The method here described is especially adapted to the Suter-Webb duplex cotton fiber sorter but with more or less obvious alteration in procedure may be carried out with two Baer sorters in tandem arrangement or with a Johannsen or other similar sorter provided with two banks of combs.

(*b*) With forceps especially constructed for this purpose, a small portion of the fibers protruding through the teeth of the comb nearest the operator shall be gripped by their free ends, gently and smoothly drawn out of the bundle, and transferred to the tips of the needles in the second field of combs, the fibers being laid straight and parallel and approximately at right angles to the face of the first comb nearest the operator, their gripped ends being released as near this comb as possible. This deposit of fibers shall be carefully pressed down into the needles of the combs with a special presser devised for the purpose.

(*c*) The process described in Paragraph (*b*) shall be continued until all the fibers are transferred, the lower combs of the first field being dropped as the work progresses. Top combs shall be inserted whenever necessary to prevent slippage of fibers not gripped by the forceps. Care shall be exercised not to bind the fibers by the use of too many top combs, nor to press the fibers too tightly into the lower field, for each will result in the breakage of fibers during the transfers.

Sorting, Second Transfer

6. The carriage of the machine shall be revolved through an angle of 180 deg. and a second transfer made as described in Section 5, except that greater care shall be used in aligning the ends of the fibers.

Arraying the Fibers

7. (*a*) The carriage of the machine shall again be revolved through an angle of 180 deg. and successive small "bites" or pulls taken from the extended tips of the fibers and placed individually in successive order and at short intervals of separation on black velvet-covered boards, the operation being continued until all the fibers in the bundle are so transferred. All pulls shall be laid on the boards as straight as possible, with the ends clearly defined to permit of easy measurement. (Careless transfers and excessive slippage of fibers from the bundle will result in pulls with ragged and uneven ends which are very difficult to measure.)

(*b*) The number of individual pulls laid on the velvet boards in a complete fiber length array shall be not less than 50 nor more than 100 for short staple cotton (less than $1\frac{1}{8}$-in. staple) and not less than 80 nor more than 125 for long staple cotton ($1\frac{1}{8}$-in. and longer staple).

Measuring

8. Beginning at the long end of the array, the lengths of the successive pulls on the velvet boards shall be measured to the nearest $\frac{1}{8}$ in. The measurement shall be recorded and the pulls classified by their mid-point lengths expressed in sixteenths of an inch. For example, $\frac{13}{16}$ in. would be the length recorded for all pulls falling within the class interval of $\frac{12}{16}$ to $\frac{14}{16}$ in., while $\frac{1}{16}$ in. would be the length of all fibers shorter than $\frac{1}{8}$ in.

Grouping and Weighing

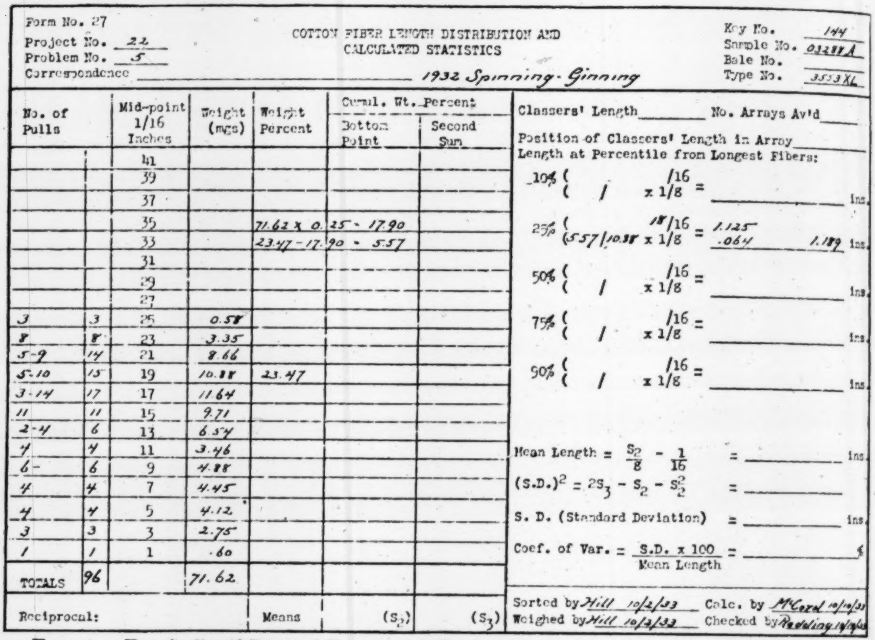
9. The pulls on the velvet boards shall be grouped according to their mid-point length, as established during measuring (see Fig. 1), and weighed to the nearest 0.05 mg. on a torsion or chemical balance graduated to 0.05 mg., and the weight of each group shall be recorded.

Calculations

10. (*a*) The weights for each length group from two or more arrays shall be added for each group. (See Fig. 2.). From these data, calculations shall be made for the percentage by weight of the fibers in each $\frac{1}{8}$-in. length interval, and for the cumulative weight percentage beginning with the longest fibers.

(*b*) The central tendency of the fiber distribution shall be expressed in terms of the upper quartile length (length at the 25 per cent point on the cumulative weight percentage-length curve beginning with the longest fibers), and may also be expressed in terms of modal, mean, median, or percentile length.

(*c*) From the basic data, also shall be calculated the statistical measures known as the mean (weight frequency) length and standard deviation; from these determinations, the coefficient of fiber length variation (the standard deviation expressed as a percentage of the mean) is calculated to indicate the degree of variability of fiber lengths or the concentration of



Form No. 27
Project No. 22
Problem No. 5
Correspondence 1932 Spinning - Ginning

COTTON FIBER LENGTH DISTRIBUTION AND
CALCULATED STATISTICS

Key No. 144
Sample No. 03288A
Bale No.
Type No. 3553XL

No. of Pulls		Mid-point 1/16 Inches	Weight (mgs)	Weight Percent	Cuml. Wt. Percent	
					Bottom Point	Second Sum
		41				
		39				
		37				
		35		71.62 x 0.	25 - 17.90	
		33		23.47 - 17.	90 - 5.57	
		31				
		29				
		27				
3	3	25	0.58			
8	8	23	3.35			
5-9	14	21	8.66			
5-10	15	19	10.88	23.47		
3-14	17	17	11.64			
11	11	15	9.71			
2-4	6	13	6.54			
4	4	11	3.46			
6-	6	9	4.88			
4	4	7	4.45			
4	4	5	4.12			
3	3	3	2.75			
1	1	1	.60			
TOTALS	96		71.62			
Reciprocal:				Means	(S_2)	(S_3)

Classers' Length ______ No. Arrays Av'd ______
Position of Classers' Length in Array ______
Length at Percentile from Longest Fibers:

10% (/16 = ______ ins.
(/ x 1/8

25% (18/16 = 1.125
(5.57/10.88 x 1/8 = .064 1.189 ins.

50% (/16 = ______ ins.
(/ x 1/8

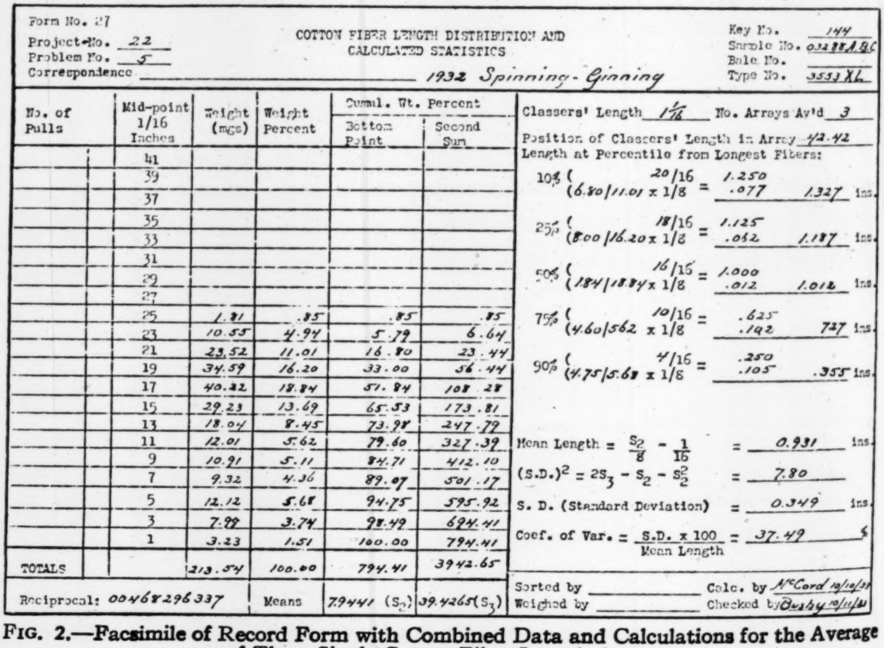
75% (/16 = ______ ins.
(/ x 1/8

90% (/16 = ______ ins.
(/ x 1/8

Mean Length = $\frac{S_2}{8} - \frac{1}{16}$ = ______ ins.
$(S.D.)^2 = 2S_3 - S_2 - S_2^2$ = ______
S. D. (Standard Deviation) = ______ ins.
Coef. of Var. = $\frac{S.D. \times 100}{\text{Mean Length}}$ = ______ %

Sorted by Hill 10/2/33 Calc. by McCord 10/10/33
Weighed by Hill 10/3/33 Checked by Redding 10/[illegible]

FIG. 1.—Facsimile of Record Form with Data and Calculations for a Single Cotton Fiber Length Array.



Form No. 27
Project No. 22
Problem No. 5
Correspondence 1932 Spinning - Ginning

COTTON FIBER LENGTH DISTRIBUTION AND
CALCULATED STATISTICS

Key No. 144
Sample No. 03288A.B.C
Bale No.
Type No. 3553XL

No. of Pulls		Mid-point 1/16 Inches	Weight (mgs)	Weight Percent	Cuml. Wt. Percent	
					Bottom Point	Second Sum
		41				
		39				
		37				
		35				
		33				
		31				
		29				
		27				
		25	1.81	.85	.85	.85
		23	10.55	4.94	5.79	6.64
		21	23.52	11.01	16.80	23.44
		19	34.59	16.20	33.00	56.44
		17	40.22	18.84	51.84	108.28
		15	29.23	13.69	65.53	173.81
		13	18.04	8.45	73.98	247.79
		11	12.01	5.62	79.60	327.39
		9	10.91	5.11	84.71	412.10
		7	9.32	4.36	89.07	501.17
		5	12.12	5.68	94.75	595.92
		3	7.99	3.74	98.49	694.41
		1	3.23	1.51	100.00	794.41
TOTALS			213.54	100.00	794.41	3942.65
Reciprocal: 00468296337				Means	7.9441 (S_2)	39.4265 (S_3)

Classers' Length 1 1/16 No. Arrays Av'd 3
Position of Classers' Length in Array 42.42
Length at Percentile from Longest Fibers:

10% (20/16 = 1.250
(6.80/11.01 x 1/8 = .077 1.327 ins.

25% (18/16 = 1.125
(8.00/16.20 x 1/8 = .062 1.187 ins.

50% (16/16 = 1.000
(1.84/18.84 x 1/8 = .012 1.012 ins.

75% (10/16 = .625
(4.60/5.62 x 1/8 = .102 727 ins.

90% (4/16 = .250
(4.75/5.68 x 1/8 = .105 .355 ins.

Mean Length = $\frac{S_2}{8} - \frac{1}{16}$ = 0.931 ins.
$(S.D.)^2 = 2S_3 - S_2 - S_2^2$ = 7.80
S. D. (Standard Deviation) = 0.349 ins.
Coef. of Var. = $\frac{S.D. \times 100}{\text{Mean Length}}$ = 37.49 %

Sorted by ______ Calc. by McCord 10/10/33
Weighed by ______ Checked by Busby 10/11/33

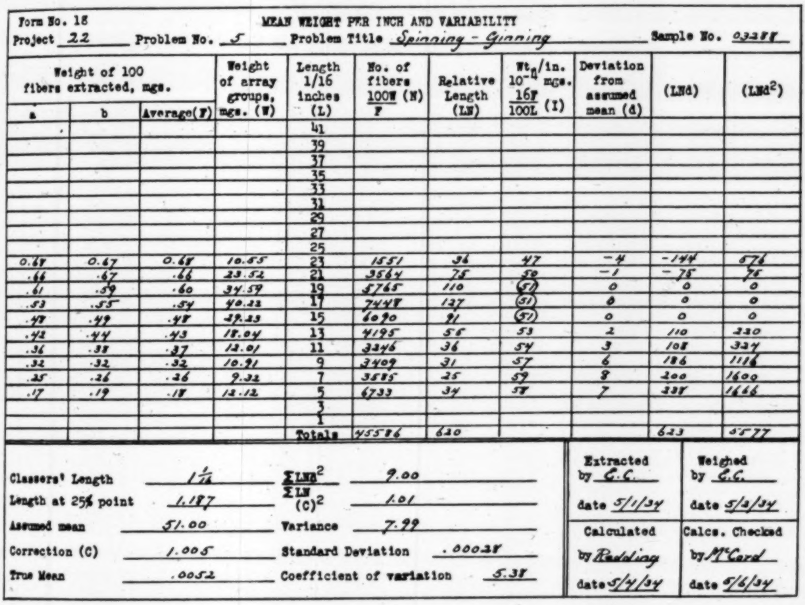
FIG. 2.—Facsimile of Record Form with Combined Data and Calculations for the Average of Three Single Cotton Fiber Length Arrays.

their frequencies. The larger the figure for the coefficient of variation, so calculated, the greater the variability of the fiber lengths in the sample.

FINENESS

Sampling

11. (*a*) The fibers for test shall be taken from not less than two length arrays prepared by the method described in Sections 3 to 9. The two or more assembled groups of corresponding length identifications shall be gently and thoroughly mixed and parallelized by successively pulling and lapping them between the fingers, the pulls in the final process being superimposed in the hand with one end of the fibers at right angles to a common base line.



Form No. 18

MEAN WEIGHT PER INCH AND VARIABILITY

Project 22 Problem No. 5 Problem Title Spinning - Ginning Sample No. 03288

Weight of 100 fibers extracted, mgs.			Weight of array groups, mgs. (W)	Length 1/16 inches (L)	No. of fibers $\frac{100W}{F}$ (N)	Relative Length (LN)	Wt_n/in. 10^{-4} mgs. $\frac{16F}{100L}$ (I)	Deviation from assumed mean (d)	(LNd)	(LNd^2)
a	b	Average(F)								
				41						
				39						
				37						
				35						
				33						
				31						
				29						
				27						
				25						
0.68	0.67	0.68	10.55	23	1551	36	47	-4	-144	576
.66	.67	.66	23.52	21	3564	75	50	-1	-75	75
.61	.59	.60	34.59	19	5765	110	(51)	0	0	0
.53	.55	.54	40.22	17	7448	127	(51)	0	0	0
.48	.49	.48	29.23	15	6090	91	(51)	0	0	0
.42	.44	.43	18.04	13	4195	55	53	2	110	220
.36	.38	.37	12.01	11	3246	36	54	3	108	324
.32	.32	.32	10.91	9	3409	31	57	6	186	1116
.25	.26	.26	9.32	7	3585	25	59	8	200	1600
.17	.19	.18	12.12	5	6733	34	58	7	238	1666
				3						
				1						
				Totals	45586	620			623	5577

Classers' Length $1\frac{1}{16}$

Length at 25% point 1.187

Assumed mean 51.00

Correction (C) 1.005

True Mean .0052

$\frac{\Sigma LNd^2}{\Sigma LN}$ 9.00

$(C)^2$ 1.01

Variance 7.99

Standard Deviation .00028

Coefficient of variation 5.38

Extracted by C.C. date 5/1/34	Weighed by C.C. date 5/2/34
Calculated by Redding date 5/4/34	Calcs. Checked by McCord date 5/6/34

FIG. 3.—Facsimile of Record Form with Data and Calculations for Determination of Cotton Fiber Fineness.

(*b*) One or more small tufts shall then be removed from the free end of the bundle and discarded. One or more thin fringes of fibers shall be withdrawn gently from the remaining portion of the bundle and laid in this condition on a board covered with black velvet.

(*c*) Two sets of 100 fibers each shall be counted out, starting at one side of the bundle and taking successive fibers.

(*d*) The operations described in Paragraphs (*a*), (*b*) and (*c*) shall be repeated for each identified length group throughout the array, with the exception of the $\frac{1}{16}$ and $\frac{3}{16}$-in. groups, and as many of those beginning at the long end of the array as may be too small to yield two sets of 100 fibers each.

Atmospheric Condition

12. All weighings shall be made in a standard atmosphere (Section 2) and not until after the samples have been conditioned for at least 2 hr. in such an atmosphere.

Weighing

13. Each group of 100 fibers shall be weighed to the nearest 0.01 mg. on a torsion or chemical balance graduated to 0.005 mg. (See Fig. 3.)

Calculations

14. (*a*) The average weight of the two corresponding groups of 100 fibers of each length identification shall be calculated and the mean fiber weight per inch determined.

(*b*) The fineness of a sample shall be the mean fiber weight per inch for the entire sample and shall be determined by mathematically weighting the weight per inch value for each length group on a basis of its contribution of fiber length to the total fiber length of the combined length arrays, and calculating the weighted mean.

(*c*) The coefficient of variation (standard deviation expressed as a percentage of the mean) of fiber weight per inch in the different length groups shall be determined, using the same frequency basis as for the mean fiber weight per inch for the sample, as in Paragraph (*b*).

IMMATURITY COUNT

Sampling

15. The test shall be made on the two sets of 100 fibers each, counted out for each length group as described in Section 11.

Mounting

16. (*a*) The fibers shall be straightened, and placed approximately parallel on a glass slide, covered with a cover slip and flooded with 18 per cent caustic soda.

(*b*) The mount shall be placed on the mechanical stage of the microscope which shall be equipped with a filar micrometer eyepiece.

Examination

17. The fibers shall be examined near their mid-portion at a magnification of approximately 400 diameters.

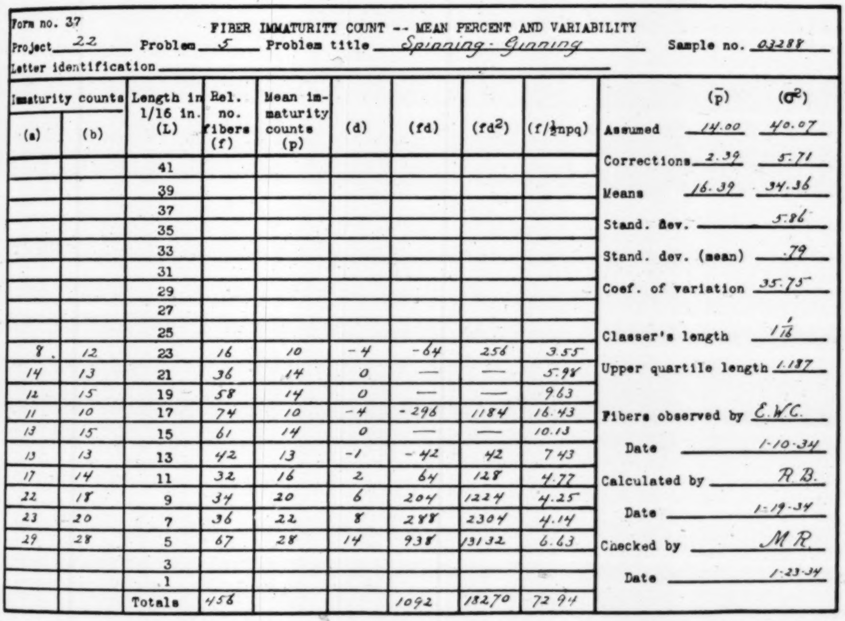
Counting and Classifying

18. (*a*) As successive fibers come into view they shall be counted, and the number of thin-walled or immature fibers in each set determined. (See Fig. 4.)

(*b*) For the purpose of this test, thin-walled or immature fibers shall be those that upon treatment with NaOH solution (18 per cent) show a fiber wall less than one-half the width of the adjacent lumen at the widest part of the fiber ribbon, that is, midway between two edge-on positions of the ribbon. All other fibers shall be classed as mature or thick-walled fibers. Doubtful classification shall be decided with the aid of the filar micrometer. (Figure 5 shows photomicrographs of thin-walled or immature fibers as compared with thick-walled fibers. The appearance of these fibers before and after treatment is shown for purposes of reference.)

Calculations

19. (*a*) The number of thin-walled or immature fibers of the two groups of 100 fibers each shall be averaged for each length group.



Form no. 37

FIBER IMMATURITY COUNT -- MEAN PERCENT AND VARIABILITY

Project 22 Problem 5 Problem title Spinning - Ginning Sample no. 03288

Letter identification

Immaturity counts		Length in 1/16 in. (L)	Rel. no. fibers (f)	Mean immaturity counts (p)	(d)	(fd)	(fd^2)	($f/\frac{1}{2}npq$)
(a)	(b)							
		41						
		39						
		37						
		35						
		33						
		31						
		29						
		27						
		25						
8	12	23	16	10	-4	-64	256	3.55
14	13	21	36	14	0	—	—	5.98
12	15	19	58	14	0	—	—	9.63
11	10	17	74	10	-4	-296	1184	16.43
13	15	15	61	14	0	—	—	10.13
13	13	13	42	13	-1	-42	42	7.43
17	14	11	32	16	2	64	128	4.77
22	18	9	34	20	6	204	1224	4.25
23	20	7	36	22	8	288	2304	4.14
29	28	5	67	28	14	938	13132	6.63
		3						
		1						
		Totals	456			1092	18270	72.94

	($\bar{p}$)	(σ^2)
Assumed	14.00	40.07
Corrections	2.39	5.71
Means	16.39	34.36
Stand. dev.		5.86
Stand. dev. (mean)		.79
Coef. of variation		35.75

Classer's length $1\frac{1}{16}$

Upper quartile length 1.187

Fibers observed by E. W. C.

Date 1-10-34

Calculated by R. B.

Date 1-19-34

Checked by M. R.

Date 1-23-34

FIG. 4.—Facsimile of Record Form with Data and Calculations for Determination of the Immaturity Count.

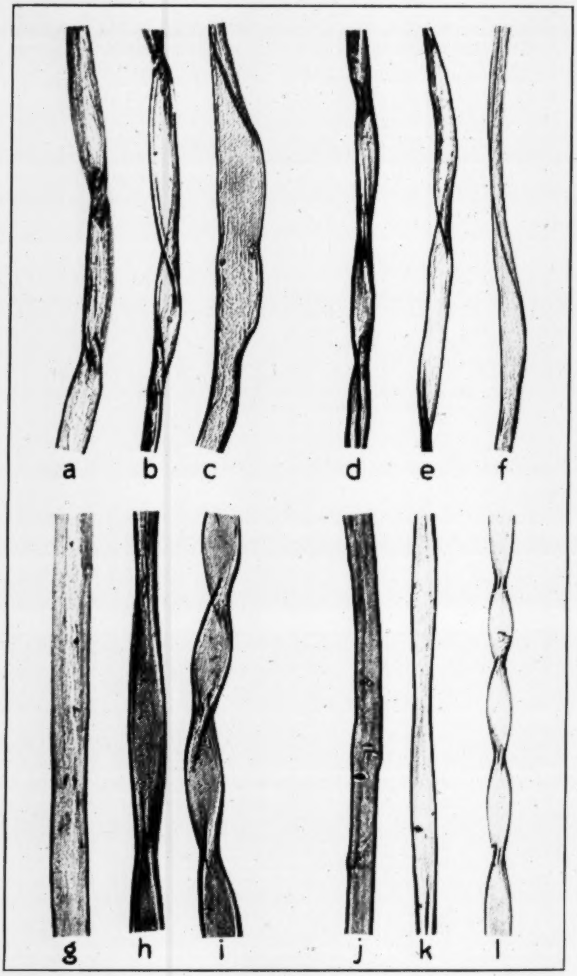
(*b*) A weighted mean of these values, based on the relative number of fibers in each length group of the combined length arrays, shall be calculated for the sample, and shall be the immaturity count. The relative number of fibers in each length group of the length array shall be calculated from the combined weight of the length group and the weight per fiber (see Sections 9 and 13).

(*c*) The coefficient of variation (standard deviation expressed as a percentage of the mean) of fiber immaturity count in the different length groups shall be determined, using the same frequency basis as for the mean immaturity count of the sample as in Paragraph (*b*).

STRENGTH

Atmospheric Condition

20. Sampling, combing, wrapping and breaking shall be performed in a standard atmosphere (Section 2) and not until after the samples have been conditioned for at least 2 hr. in such an atmosphere.



Courtesy of *U. S. Department of Agriculture*

FIG. 5.—Coarse and Fine Fibers of Different Degrees of Maturity Before and After Treatment with Caustic Soda Solution (× 170).

Specimen *a* represents a thick-walled fiber while *b* and *c* represent thin-walled fibers from a *coarse* American upland cotton, staple length $\frac{3}{4}$ in.; *g*, *h*, and *i* are the same fibers respectively as *a*, *b*, and *c* after treatment with 18 per cent caustic soda solution.

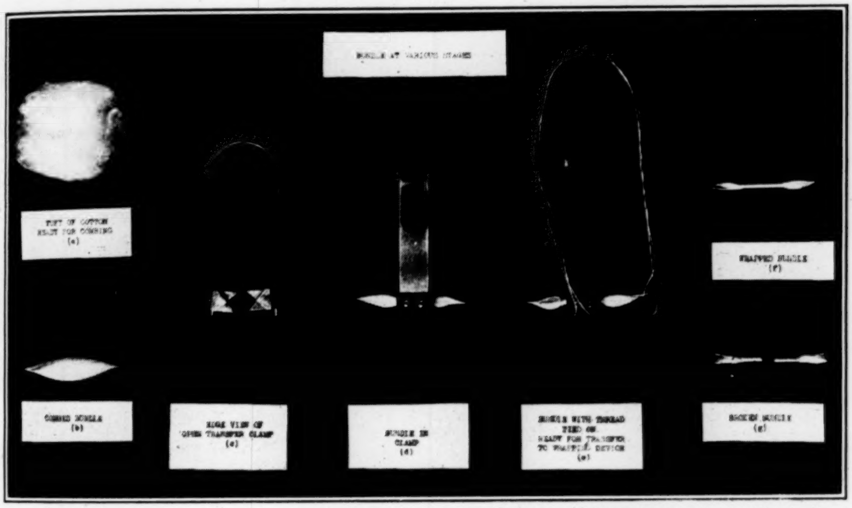
Specimen *d* represents a thick-walled fiber while *e* and *f* represent thin-walled fibers from a *fine* American upland cotton, staple length $1\frac{3}{8}$ in.; *j*, *k*, and *l* are the same fibers respectively as *d*, *e*, and after treatment with 18 per cent caustic soda solution.

Sampling

21. (*a*) A small handful of cotton shall be removed carefully from the original sample (see Note, Section 3 (*a*)), divided into two approximately equal portions by pulling apart in the hands, and the portions superimposed with their broken surfaces coinciding.

(*b*) From the broken surface of the sample, three to four pulls shall be drawn out with the hand and combined. (See Fig. 6 (*a*).)

(*c*) The operations described under Paragraphs (*a*) and (*b*) shall be repeated for each of the ten bundles required for the complete test.


BUNDLE AT VARIOUS STAGES
TUFT OF COTTON READY FOR COMBING (a)
COMBED BUNDLE (b)
SIDE VIEW OF OPEN TRANSFER CLAMP (c)
BUNDLE IN CLAMP (d)
BUNDLE WITH THREAD TIED ON READY FOR TRANSFER TO TRAPPING DEVICE (e)
TRAPPED BUNDLE (f)
BROKEN BUNDLE (g)

Courtesy of *U. S. Department of Agriculture*

FIG. 6.—Stages in the Preparation of the Chandler Bundles.

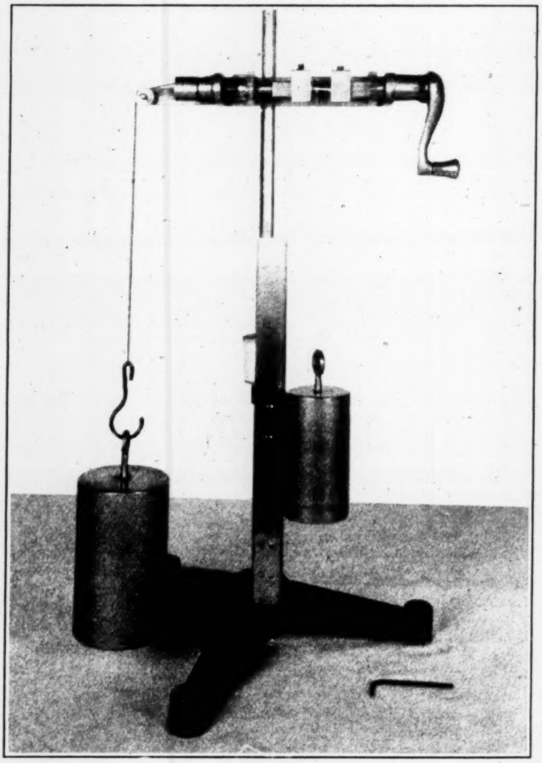
Combing

22. (*a*) Two stationary combs shall be used, a coarse comb about 2.5 in. in width with needles 0.025 in. in diameter at the base, 0.5 in. in length, and 16 needles to the inch, and a fine comb 2.0 in. in width with needles 0.015 in. in diameter at the base, 0.5 in. in length and, 48 needles to the inch.

(*b*) The fibers shall be carefully straightened and paralleled by gripping the tuft of fibers (see Fig. 6 (*a*)) near its center with the thumb and forefinger and combing thoroughly. The outer edge of the tuft shall be drawn through the coarse comb and the action gradually advanced toward the center of the tuft, using fifteen strokes through the comb. The tuft then shall be reversed, the combed end grasped about $\frac{1}{8}$ in. inside of the combed position, and the other end combed fifteen times, the combings being discarded.

(*c*) The roughly prepared bundle shall be more completely combed by drawing each end through the fine comb 20 times, after which the bundle shall be adjusted in size by separating as many fibers from the side as may be necessary to give a wrapped bundle with a corrected circumference within the range 0.115 to 0.135 in. (The correct range of circumference may easily be attained with the aid of a torsion balance of a 50-mg. capacity.)

(*d*) The bundle then shall be finished on the fine comb by stroking each end fifteen times.



Courtesy of *U. S. Department of Agriculture*

FIG. 7.—Wrapping Device with Bundle in Jaws and Weights Suspended.

Wrapping

23. (*a*) Each bundle shall be wrapped by means of a special wrapping device and process. (See Fig. 7).

(*b*) The bundle shall be inserted in a special clamp (see Fig. 6 (*c*), (*d*), and (*e*)), and the fibers straightened by gently pulling on each end of the bundle.

(*c*) The two ends of about 20 in. of No. 20 sewing thread shall be tied to opposite ends of the bundle near the sides of the clamp. (See Fig. 6 (*e*).)

(*d*) The bundle, still in the clamp, shall be transferred to and fastened securely in the wrapping device.

(*e*) The bundle shall be stretched lengthwise by tension from a 10-lb. weight suspended over a pulley. The bundle clamp then shall be removed and a 4-lb. weight suspended and allowed to hang freely in the loop of the wrapping thread.

(*f*) The handle of the wrapping device shall be turned about a dozen turns, care being exercised to hold the two threads slightly apart with the fingers of one hand in order that the thread will pack closely during successive turns of the bundle. The handle shall be stopped at an index on its shoulder and the position of the weight hanging freely in the thread shall be recorded. (See Fig. 8.)

TABLE I.—CORRECTIONS APPLICABLE TO MACHINE BREAKS FOR COTTONS HAVING DIFFERENT MEAN CORRECTED MACHINE BREAKS[a] AND CIRCUMFERENCES DEVIATING FROM STANDARD CIRCUMFERENCE.[b,c]

Deviation of Circumference from Standard, in.	Corrections in Pounds Applicable to Cotton Having Average Corrected Machine Break of														
	60 lb.	65 lb.	70 lb.	75 lb.	80 lb.	85 lb.	90 lb.	95 lb.	100 lb.	105 lb.	110 lb.	115 lb.	120 lb.	125 lb.	130 lb.
0.001.......	0	1	1	1	1	1	1	1	1	1	1	1	1	2	2
0.002.......	1	1	1	1	2	2	2	2	2	2	3	3	3	3	3
0.003.......	1	2	2	2	2	3	3	3	3	4	4	4	4	5	5
0.004.......	2	2	3	3	3	4	4	4	5	5	5	6	6	6	7
0.005.......	2	3	3	4	4	4	5	5	6	6	7	7	7	8	8
0.006.......	3	3	4	4	5	5	6	6	7	7	8	8	9	9	10
0.007.......	3	4	4	5	6	6	7	7	8	9	9	10	10	11	12
0.008.......	4	4	5	6	6	7	8	8	9	10	10	11	12	12	13
0.009.......	4	5	6	6	7	8	9	9	10	11	12	13	13	14	15
0.010.......	5	5	6	7	8	9	10	11	11	12	13	14	15	16	16

[a] The mean corrected machine break is obtained by a first approximation, by allowing 1 lb. for each 0.001-in. deviation of the mean circumference from standard circumference (0.125 in.). This determines the proper column for the corrections.

[b] Bundles smaller than standard circumference require that the corrections be added; those larger than standard require that the corrections be subtracted.

[c] The corrections are calculated by means of the formula $R = 0.01690\ Bs - 0.55$; where Bs is the mean machine break for bundles of standard circumference, and R is the correction for each 0.001-in. deviation of circumference from standard.

(*g*) The wrapping shall be continued exactly ten revolutions of the handle, the threads again being held apart slightly, as before. The fingers then shall be removed from the thread and the new position of the suspended weight recorded.

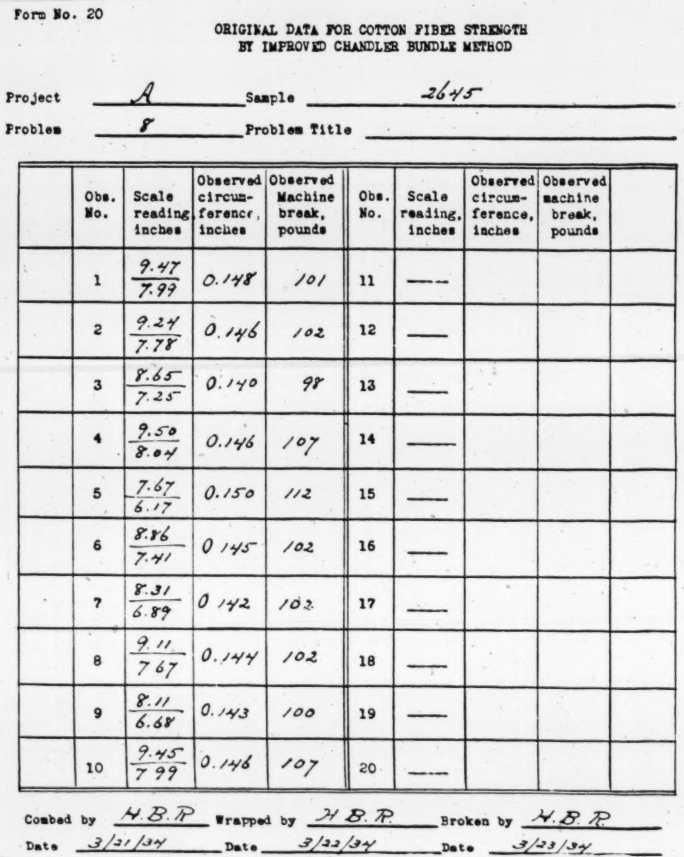
(*h*) The wrapping shall be continued until the threads meet near the center of the bundle and reverse their course along the bundle. A pencil mark shall be made where the two threads meet. Finally, wrapping shall be continued until the threads reach the sides of the wrapping jaws.

(*i*) The two weights shall be removed. The loop of the thread shall be cut in the center and each end tied around its respective bundle end.

Breaking

24. (*a*) Breaking jaws specially designed and constructed for the purpose shall be used in this test. The essential feature of the jaw is a slot into which is pressed a piece of hard sole leather. A lengthwise groove is made in the center of the leather by partly impressing a wire of $\frac{1}{16}$ in. in diameter.

(*b*) With the special jaws (Paragraph (*a*)) set so that they are in



Form No. 20

ORIGINAL DATA FOR COTTON FIBER STRENGTH
BY IMPROVED CHANDLER BUNDLE METHOD

Project A Sample 2645

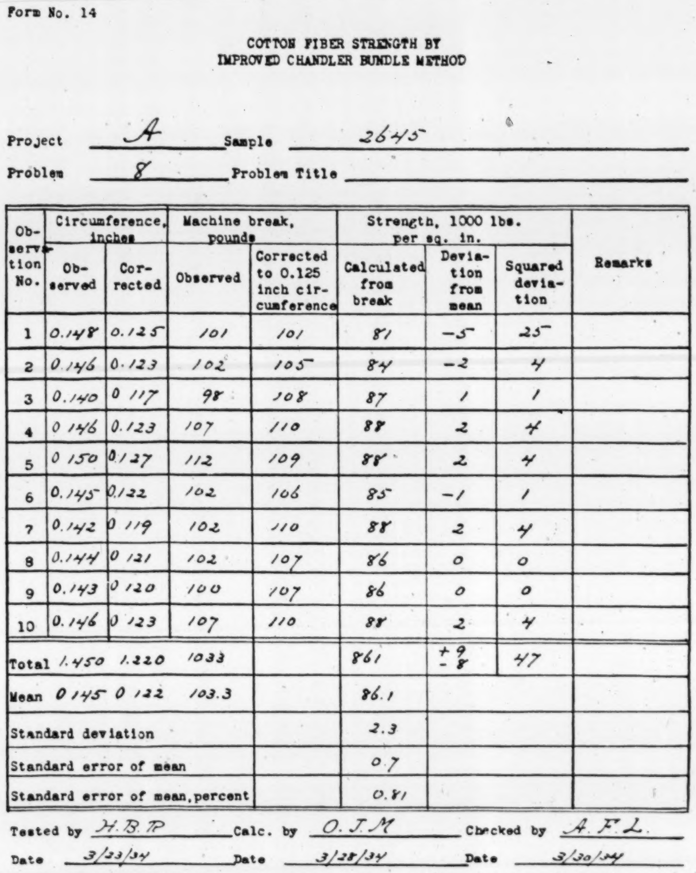
Problem 8 Problem Title

	Obs. No.	Scale reading inches	Observed circumference, inches	Observed Machine break, pounds	Obs. No.	Scale reading, inches	Observed circumference, inches	Observed machine break, pounds	
	1	9.47/7.99	0.148	101	11	----			
	2	9.24/7.78	0.146	102	12	----			
	3	8.65/7.25	0.140	98	13	----			
	4	9.50/8.04	0.146	107	14	----			
	5	7.67/6.17	0.150	112	15	----			
	6	8.86/7.41	0 145	102	16	----			
	7	8.31/6.89	0 142	102	17	----			
	8	9.11/7 67	0.144	102	18	----			
	9	8.11/6.68	0.143	100	19	----			
	10	9.45/7 99	0.146	107	20	----			

Combed by H.B.R. Wrapped by H.B.R. Broken by H.B.R.

Date 3/21/34 Date 3/22/34 Date 3/23/34

FIG. 8.—Facsimile of Record Form Used in the Modified Chandler Bundle Method and Data Representing the Observed Scale Readings and Machine Breaks.



Form No. 14

COTTON FIBER STRENGTH BY
IMPROVED CHANDLER BUNDLE METHOD

Project A Sample 2645

Problem 8 Problem Title

Observation No.	Circumference, inches		Machine break, pounds		Strength, 1000 lbs. per sq. in.			Remarks
	Observed	Corrected	Observed	Corrected to 0.125 inch circumference	Calculated from break	Deviation from mean	Squared deviation	
1	0.148	0.125	101	101	81	−5	25	
2	0.146	0.123	102	105	84	−2	4	
3	0.140	0 117	98	108	87	1	1	
4	0 146	0.123	107	110	88	2	4	
5	0 150	0.127	112	109	88	2	4	
6	0.145	0.122	102	106	85	−1	1	
7	0.142	0 119	102	110	88	2	4	
8	0.144	0 121	102	107	86	0	0	
9	0.143	0 120	100	107	86	0	0	
10	0.146	0 123	107	110	88	2	4	
Total	1.450	1.220	1033		861	+9 −8	47	
Mean	0 145	0 122	103.3		86.1			
Standard deviation					2.3			
Standard error of mean					0.7			
Standard error of mean, percent					0.81			

Tested by H.B.R. Calc. by O.J.M. Checked by A.F.L.

Date 3/23/34 Date 3/28/34 Date 3/30/34

FIG. 9.—Facsimile of Record Form Used for Recording the Corrected Circumference and Machine Break and for Calculating the Strength and Variability of Strength per Square Inch.

contact, each bundle shall be placed in them so that the mark near the center of the bundle coincides with the point of contact of the jaws.

(*c*) Each bundle, so clamped in the jaws, shall be placed in a tension testing machine with a capacity of 300 lb. and the bundle broken with the pulling jaws moving at the rate of 12 in. ± $\frac{1}{2}$ in. per min. Only a break that is sharp and at the mark on the bundle shall be considered to be successful.

Calculation

25. (*a*) The corrected circumference shall be calculated by obtaining the difference in the two scale readings, dividing this result by ten, and then subtracting 0.023 in. (correction for thread diameter). (See Fig. 9.)

(*b*) Each machine break shall be corrected to a bundle of standard size (0.125-in. circumference) by recourse to the accompanying Table I. The proper column to use in Table I is determined by calculating the average of the ten machine breaks and the average of their corresponding circumferences and adding or subtracting 1 lb. for each deviation of 0.001 in. according as the average size is smaller or larger, respectively, than standard. If the average circumference deviates more than 0.001 in. from the standard, a second approximation shall be made.

(*c*) The strength per square inch of each bundle shall be obtained by dividing the corrected machine break by the cross-sectional area (0.0012434 sq. in.) of a bundle of standard size. (This may be conveniently obtained by reference to a prepared table or chart.)

(*d*) The strength of a sample shall be the average strength per square inch as obtained from ten successful breaks.

TENTATIVE METHODS OF TESTING AND TOLERANCES

FOR

RAYON[1]

A.S.T.M. Designation: D 258 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1926; REVISED, 1927, 1931, 1932, 1935, 1937.

Scope

1. These methods of testing and tolerances apply to the various types of rayon in the form of filaments or yarn.

DEFINITIONS

Rayon

2. (*a*) *Rayon.*—A generic term for filaments made from various solutions of modified cellulose by pressing the cellulose solution through an orifice, and solidifying it in the form of a filament.

NOTE.—Rayon as commercially manufactured is produced by four different processes. The resulting rayons from each process while similar in appearance differ among themselves and with each other to a greater or lesser degree according to the differences in their physical and chemical properties. The different type rayons, as defined in these specifications, are known as follows:

(*a*) Nitrocellulose Rayon (Chardonnet)
(*b*) Viscose Rayon
(*c*) Cuprammonium Rayon
(*d*) Cellulose-Acetate Rayon

(*b*) *Rayon Yarns.*—Yarn composed of more than one continuous rayon filament.

(*c*) *Spun Rayon.*—Yarn made from cut rayon filaments, the cut filaments being drawn out and twisted into a yarn by usual spinning processes, as follows:

(*1*) Cotton system of spinning (ring and mule).
(*2*) Worsted system of spinning (modified Bradford) (cap or ring).
(*3*) Spun-silk method of spinning (ring or mule).

Classification

3. (*a*) *Nitrocellulose Rayon.*—Filaments composed of a regenerated or denitrated cellulose which has been coagulated or solidified from a solution of nitrated cellulose.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

(*b*) *Viscose Rayon.*—Filaments composed of a regenerated cellulose which has been coagulated or solidified from a solution of cellulose xanthate.

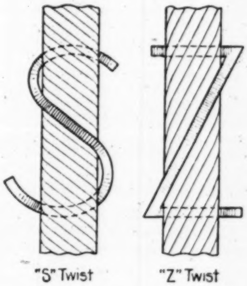
(*c*) *Cuprammonium Rayon.*—Filaments composed of a regenerated cellulose which has been coagulated or solidified from a solution of cellulose in ammoniacal copper oxide.

(*d*) *Cellulose-Acetate Rayon.*—Filaments composed of an acetic ester of cellulose which has been coagulated or solidified from its solution.

Direction of Twist

4. The direction of twist shall be as defined in the Tentative Definitions and Terms Relating to Textile Materials (A.S.T.M. Designation: D 123 - 37 T) of the American Society for Testing Materials, as follows:

Twist, Direction of.—A yarn or cord has **S** twist if, when held in a vertical position, the spirals conform in slope to the central portion of the letter "S," and **Z** twist if the spirals conform in slope to the central portion of the letter "Z."



Denier (Rayon Yarn)

5. The denier of a rayon yarn is numerically equal to the weight in grams of 9000 meters.

Number (Spun Rayon Yarn)

6. Spun rayon yarns are numbered on the basis of the system by which they are spun.

TOLERANCES

Tolerances

7. Tolerances shall be the limits within which a textile must come in its specified characteristics in order that it shall constitute a good delivery on contract.

Denier of Rayon Yarn

8. (*a*) The average denier of the rayon yarn, either bleached or unbleached, in each skein, tube, spool, cop, pirn, or cone of rayon as supplied by the seller, as found by test, shall not vary above or below the specified denier more than:

10 per cent for denier finer than 150, and
8 per cent for 150 denier or coarser.

(*b*) The average denier of the rayon yarn in the singles, either bleached or unbleached, in each case of skeins, tubes, spools, cops, pirns, cones, or a beam warp as found by test shall not vary above or below the specified denier more than:

5 per cent for denier finer than 150, and
4 per cent for 150 denier or coarser.

Number of Spun Rayon Yarn

9. The average number of the spun rayon yarn in the singles, either bleached or unbleached, in each case of skeins, tubes, spools, cops, pirns, cones, or a beam warp, as found by test, shall not vary more than 5 per cent above or below the specified number. Spun rayon yarns are numbered on the basis of the system by which they are spun.

Tensile Strength

10. (*a*) The average tensile strength of the rayon yarn in each case of skeins, tubes, spools, cops, pirns, cones, or a beam warp in the singles or plied, either bleached or unbleached, as found by test, shall not be less than the specified tensile strength.

(*b*) Tensile strength at break, or at the yield point, may be specified. Tensile strength shall be expressed as the number of grams per denier of the yarn before testing.

Twist

11. The average twist of the rayon yarn in each case of skeins, tubes, spools, cops, pirns, cones, or a beam warp shall not vary beyond the following specified limits:

TURNS PER INCH	PERMISSIBLE VARIATIONS
Under 7½	± 25 per cent
7½ to 10	± 15 per cent
Over 10	± 5 per cent

METHODS OF TESTING

IDENTIFICATION

General

12. (*a*) The following procedure assumes that the rayon is known to be one of the four common types of rayon defined in Section 3. If this is not definitely known, the sample shall be examined according to the general procedure given in Tentative Methods for the Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles (A.S.T.M. Designation: D 276 – 37 T) of the American Society for Testing Materials.[1]

(*b*) Three methods of identification are given, depending on physical properties, chemical properties and optical properties. In general, no one method is completely reliable, particularly when distinguishing between cuprammonium and viscose rayon.

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[1] See p. 1174.

(*c*) While not essential, it is desirable that the analyst should have available a series of authentic samples of the different types of rayon yarn. These samples should be from various sources and should be kept as nearly up to date as possible.

Preparation of Sample

13. These methods are intended for rayon which is substantially free from color imparted by dyestuffs or colored pigments either before or after spinning, is in either bleached or unbleached state (Note), and is free from oil, finish or other foreign substances. If any of the latter are present, they shall be removed from the sample by digesting successively with the following solvents:

(*a*) Distilled water at a temperature of 158 to 176 F. (70 to 80 C.) for 15 min.

(*b*) Denatured alcohol at room temperature for 5 min.

(*c*) Diethyl ether (sulfuric ether) at room temperature for 5 min.

The sample shall be wrung out after each immersion in order to remove the excess of the solvent before transferring it to the next solvent. Finally, the sample shall be allowed to dry at room temperature.

NOTE.—The procedures described herein may be applied to colored yarn, stripped or unstripped, but the results are less reliable. The methods given in this section for removing "other foreign substances" are not suitable for complete removal of materials insoluble in these solvents.

Physical Tests

14. (*a*) A few strands of the sample shall be twisted and the mass brought as close as possible to a flame without permitting it to burn. Cellulose-acetate rayon is thermoplastic and will soften or melt, depending on the temperature. In the latter case it forms a hard ball. Nitrocellulose, viscose and cuprammonium rayon do not fuse when heated.

(*b*) If sufficient sample of rayon yarn is available, the denier and the filament count shall be determined. These values shall be compared with the various numbers offered for sale commercially by the different manufacturers of rayon.

NOTE.—This test is valuable only when the sample is known to be material of fairly recent manufacture and is known to be a commercial product, not an experimental lot. Occasionally, this will definitely determine the type of rayon yarn. At other times it merely limits the number of possibilities, and on occasion, is of no assistance.

Chemical Examination

15. (*a*) *To Identify Cellulose-Acetate Rayon:*

Treat a few strands of the sample with c.p. acetone. Cellulose-acetate rayon dissolves.

Treat a few strands of the sample with a boiling solution of acetic acid (40 g. of glacial acetic acid with 60 g. of distilled water). Cellulose-acetate rayon dissolves.

Nitrocellulose and cuprammonium and viscose rayon do not dissolve in either of the two solvents mentioned above. Partially saponified or hydrolyzed cellulose-acetate rayon may be partly or completely insoluble in the solvents described above.

(*b*) *To Identify Nitrocellulose Rayon:*

If the sample is not cellulose-acetate rayon, treat a few strands with diphenylamine reagent (66 g. of concentrated sulfuric acid, 33 g. of glacial acetic acid, 1 g. of diphenylamine) (Note 1). Nitrocellulose rayon gives a blue color; cuprammonium and viscose rayon do not give a blue color (Note 2). Nitrocellulose rayon dissolves faster than cuprammonium or viscose rayon.

NOTE 1.—Diphenylbenzidine may be substituted for diphenylamine in the above reagent.

NOTE 2.—This is essentially a test for oxidizing agents and cannot be relied on in the presence of residual bleaches such as peroxide or hypochlorite. A reagent which is exposed to the air for some time will develop a blue shade in the absence of other oxidizing agents. Active reducing agents such as hydrosulfite and some dyestuffs interfere with the test.

(*c*) *To Identify Viscose Rayon:*

If the sample is not cellulose-acetate or nitrocellulose rayon, make the following sulfide test:

Place a 5-g. sample (Note 1) of the rayon to be tested in a 250-ml. Erlenmeyer flask together with 100 ml. of water and 3 ml. of concentrated sulfuric acid. Completely cover the mouth of the flask with a piece of lead acetate paper, fastening the paper securely in position and allow to stand at a temperature of 194 to 203 F. (90 to 95 C.) for 4 hr. Viscose rayon stains the lead acetate paper yellowish to brown or possibly black (Note 2). Cuprammonium rayon does not discolor the paper.

NOTE 1.—If less than 5 g. of the sample is available take a smaller sample with the same relative quantities of reagent and place them in a test tube.

NOTE 2.—This is a test for sulfides. As the methods for purifying viscose rayon become more and more efficient, samples are encountered occasionally which do not give a positive test.

(*d*) *To Identify Cuprammonium Rayon by Stain Tests:*

Four different stain tests are recommended as follows because no one stain test can be relied upon in every case:

Wright Stain Test.—Immerse an air-dry sample for a few seconds in a boiling saturated alcoholic solution of Wright stain, rinse thoroughly in cold water. Viscose rayon is stained blue; cuprammonium rayon is stained violet.

Erie Fast Orange.—Dye the sample in a 0.2 per cent solution of Erie Fast Orange CG (Color Index No. 621). Viscose rayon is white to weak orange; cuprammonium rayon is dyed deep orange.

Neocarmin W.—Immerse a small sample for 3 to 5 min. in a solution of Neocarmin W (Eimer & Amend). Rinse in running water, pass quickly through water containing a trace of ammonia, rinse well and dry. Viscose rayon is dyed violet; cuprammonium rayon is dyed blue.

Diphenyl Fast Green.—Immerse a small sample in a 1 per cent solution of Diphenyl Fast Green B (Geigy Co.) for 2 min. at 68 F. (20 C.) Add Glauber's salt. Remove, rinse and dry. Cuprammonium rayon is dyed green, viscose rayon is undyed.

NOTE.—Dye tests depend upon the nature of the surface of the rayon yarn and the subsequent treatment received in processing and finishing. While generally indicative, manufacturing processes occasionally mask the difference between these two types of rayon and it is desirable to supplement the above tests with a microscopical examination.

Cuprammonium rayon cannot be satisfactorily identified by determining residual copper.

If previous tests indicate that the sample is not viscose, acetate or nitrocellulose rayon, it may be presumed that it is cuprammonium rayon but this should be checked by physical and optical methods.

Optical Test Apparatus

16. When possible, a microscopic examination of the lateral surface and cross-section of the sample should be made, particularly in the case of cuprammonium and viscose rayon. For this purpose any good chemical microscope may be used, but examination may be made with the following equipment which represents the least amount of apparatus which will give satisfactory results:

Microscope with achromatic objectives 16 mm. and 4 mm., 10× eye piece, Abbe Condenser N. A. 120.

The following accessories will be required:

A lamp with Daylite glass.

Microscope slide and cover glasses.

Mounting medium, either methylene iodide or monobrom naphthalene.

Optical Tests

17. (*a*) *Examination of the Lateral Surface.*—Clean the microscope slide by flaming or with solvent, place on this a few filaments from the unknown sample and add a drop of the mounting medium to the filaments on the slide. Place a cover glass over the sample and liquid, pressing them down until the mounting medium wets the whole of the underside of the cover glass. Place the slide on the microscope stage, adjust the light, and focus, using first the 16 mm. and then the 4 mm. objective with the 10× eye piece. Compare the samples with the series of standard plates appended.[1]

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[1] See the Photomicrographs of Common Textile Fibers in the Appendix I, pp. 1213 to 1217.

Viscose rayon can be readily distinguished from cuprammonium rayon by the many fine striations parallel with the axis of the filaments. Cuprammonium rayon is unstriated.

(*b*) *Cross-Sectional Examination.*—Prepare a thin cross-section of the sample by twisting several strands together and embedding them in wax, in a cork, or in a suitable sectioning device. Cut a thin section by means of a microtome or a sharp safety razor blade. Mount the sample on a microscope slide and if wax has been used, remove this by dissolving with xylene. Focus as described above and compare the image with the series of standard cross-section plates appended.[1]

Viscose rayon usually has an irregular serated edge, cuprammonium rayon is almost round and has a smooth unserated edge.

NOTE.—Certain types of high tenacity viscose rayon processed in strong acid baths (Lilienfeld type) appear much more like cuprammonium rayon than regular viscose rayon.

YARN DENIER

Yarn Denier Determination

18. (*a*) The denier of rayon yarn shall be determined (except when the rayon is on beams, see Paragraph (*b*)) from skeins which have been prepared, reeled, and weighed under prevailing atmospheric conditions from the spools, cops, tubes, cones, skeins, or other packages.

(*b*) Rayon received on beams shall be tested as specified.

Test Skein

19. A test skein of 200 turns of yarn shall be reeled, using any reel employing a straight wind traverse having a perimeter of 112.5 cm. (Note.) For yarns in skein form, a speed of 100 to 150 r. p. m. shall be used. For yarns on spools, cops, tubes or cones, the yarn shall be drawn off over end and a speed of 200 to 300 r. p. m. of the reel shall be used. The tension on the yarn shall not be heavier than is necessary to lay the yarn smoothly on the reel at the specified speed. The skeins shall be weighed separately on a balance which shall be accurate to 0.25 per cent of the average weight of one skein.

NOTE.—In a laboratory which is equipped for testing cotton yarns only, the skeins may be prepared on any reel having a perimeter of 1.5 yd. Each skein may contain 200 turns as before if strength tests are to be subsequently made on it.

If a skein of this length is used, the yarn denier shall be calculated from the corrected weight (Section 22) by the following formulas:

When weighing is made in grams:

$$\text{Denier} = \frac{\text{Weight of 300-yd. skein in grams} \times 1.64}{\text{0.05 gram (per denier)}}$$

When skeins are weighed in grains:

$$\text{Denier} = \frac{\text{Weight of 300-yd. skein in grains} \times 1.64}{\text{0.7716 grain (per denier)}}$$

When test skeins of 200 turns of yarn are not required for the strength test, the length of skeins reeled on the cotton reel may be 246 yd. (225 m.), or 492 yd. (450 m.) if desired. These skeins shall be weighed in accordance with Section 19.

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[1] See the Photomicrographs of Common Textile Fibers in the Appendix I, pp. 1213 to 1217.

Number of Denier Tests

20. Two skeins of 200 turns each from each of 10 spools, cops, tubes, cones or skein bundles, from one case out of every 10 cases shall be made, and the average of these 20 tests shall be the denier of the rayon yarn.

Weight Correction

21. The weight of each skein shall be reduced to a common basis of standard moisture regain for the class of rayon under test by the following formulas:

(*a*) *For nitrocellulose, viscose and cuprammonium rayons:*

$$\text{Weight, corrected to 11 per cent moisture regain} = \frac{\text{Actual skein weight} \times 111}{100 + \text{actual percentage of regain}}$$

(*b*) *For cellulose acetate rayons:*

$$\text{Weight, corrected to 6.5 per cent moisture regain} = \frac{\text{Actual skein weight} \times 106.5}{100 + \text{actual percentage of regain}}$$

Calculation

22. The denier shall be calculated as follows:

$$\text{Denier} = \text{Corrected weight of 225-meter skein in grams} \times 40$$

23. The yardage per pound of rayon of any given denier may be calculated by the following formula:

$$\text{Yards per pound of rayon} = \frac{\text{4,464,528 (yards per pound of one denier yarn)}}{\text{Given denier}}$$

Moisture Regain, Actual

24. To determine the actual percentage of moisture regain present in the test skeins (Section 19), two groups of three skeins each shall be taken immediately after weighing and the weight of each group recorded. They shall then be placed in two separate baskets in a conditioning oven and dried to constant weight on a balance sensitive to 0.25 per cent of the average weight of one skein at 220 to 230 F. (105 to 110 C.). The moisture regain of each group shall then be calculated as the percentage of the dry weight and the average of these two shall be the actual percentage of moisture regain in the skeins.

STRENGTH

(*A*) *Preferred Method*

Test Skeins

25. One skein from each of the ten spools, cops, tubes, cones or skeins drawn for a sample shall be prepared as described in Sections 18 and 19. The number of turns in these strength skeins shall be as specified in Table I. For deniers which require 200 ends, ten of the test skeins (Section 20), may be used if desired. Denier skeins to be used for the strength test shall be removed from the reel *after* collapsing the reel sufficiently to prevent stretching or damage, before being placed in the tensile testing machine.

Table I.

Specified Denier of Rayon	Number of Turns in Strength Skein
Finer than 51 denier	400
51 to 76 denier	200
76 to 121 denier	150
121 to 201 denier	100
201 denier and coarser	50

Conditioning

26. (*a*) The test skeins shall be conditioned to a moisture equilibrium in an atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of ±2 per cent is permitted in relative humidity and +10 F. (6 C.) in temperature.

(*b*) It shall be considered that moisture equilibrium is reached when, after free exposure to the atmosphere kept in rapid motion by an electric fan, there is no progressive increase in weight. Moisture equilibrium shall be approached from the dry side (not moisture free).

Note.—It is believed that test skeins will reach moisture equilibrium after such exposure for a 3-hr. period.

Apparatus

27. (*a*) *Machine.*—The test skeins shall be broken on a tensile testing machine of the pendulum type of 25-kg. (55-lb.) or 50-kg. (110-lb.) capacities. The pulling jaw shall travel at a speed of 6 in. per min.

(*b*) *Recording Device.*—The machine shall be equipped with an automatic charting device to record stretch at any load.

(*c*) *Jaws.*—Clamps for holding the test skeins shall consist of flat metallic jaws. These shall be covered with one layer of rubber tire tape which shall press directly against the specimen. One gripping surface shall be hinged or swiveled and the other shall be rigidly connected to the frame of the jaw. The pressure between the jaws shall be secured by any suitable mechanical device so constructed as to grip the yarns firmly before the testing load is applied and prevent slipping during the progress of the test.

(*d*) Any tendency to friction, backlash, or play in the recording device, lower jaw, or screw, shall be overcome as far as practicable by counter balancing.

Procedure

28. The conditioned test skeins shall be separately broken on the testing machine. The initial distance between jaws shall be 10 cm. (4 in.). The skein to be tested shall first be clamped in the upper jaws, spread out evenly so that the ends are parallel forming a ribbon approximately $\frac{3}{8}$ to $\frac{3}{4}$ in. in width. The rayon shall then be drawn down through the lower jaws and spread out to make a band of equal width, pulled just taut and clamped. The lower capacity machine shall be used until the swing of the pendulum

exceeds an angle of 45 deg. from the vertical. When the yarns break above this mark, the higher capacity of testing machine shall be used.

Number of Strength Tests

29. One test shall be made on each of the 10 skeins prepared as described in Section 25 and the average of these 10 tests shall be the tensile strength.

B. *Alternate Method*

Apparatus

30 (*a*) A single strand tester of proper capacity with the jaws set 18 in. between grips shall be used.

(*b*) The speed of the pulling jaw of a pendulum type testing machine shall be 12 ± ½ in. per min.

(*c*) The speed of operation of a constant specimen-rate-of-load type testing machine shall be such that the rate of application of the load shall be 4 g. per denier per min.

Procedure, Alternate Method

31. Five single strands from each of 10 spools, cops, tubes, cones, or skeins shall be broken after conditioning the package in accordance with Section 26.

NOTE.—It is believed that the packages will reach moisture equilibrium after conditioning in accordance with Section 26 for a 3-hr. period for skeins and for a 12-hr. period for any other form of package.

Number of Strength Tests

32. The average of 50 tests free from breaks at the jaws shall be the strength.

TWIST

Twist Specimen

33. The specimen of yarn for twist determination shall be drawn from the side of the package instead of over the end in order to avoid changing the twist.

Twist Test Procedure

34. The twist shall be determined in any standard twist tester with jaws set 10 in. apart. The yarn shall be clamped in the jaws under a definite tension by attaching a weight. The tension weight to be used shall be as follows:

YARN	TENSION WEIGHT
75 denier and finer	10 g.
150 denier to 75 denier	20 g.
Coarser than 150 denier	30 g.

NOTE.—Where yarn is unraveled from cloth for test, it is recommended that the tension weight specified be increased by 10 g. in each classification.

Twist Calculation

35. Two samples of yarn from each of five spools, cops, tubes, cones, or skeins shall be tested and the average of these 10 tests calculated to turns per inch shall be the twist.

MOISTURE REGAIN

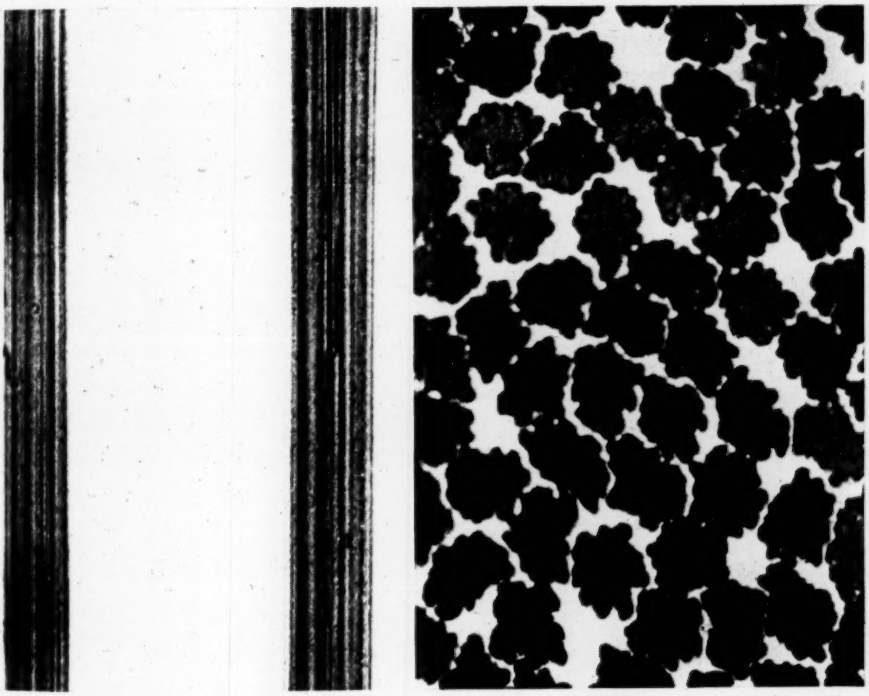
Moisture Regain

36. The standard moisture regain of nitrocellulose, viscose and cuprammonium rayons shall be 11 per cent of the weight of the moisture-free yarn.

37. The standard moisture regain of cellulose-acetate rayons shall be 6.5 per cent of the weight of the moisture-free yarn.

APPENDIX

PHOTOMICROGRAPHS OF RAYON FIBERS

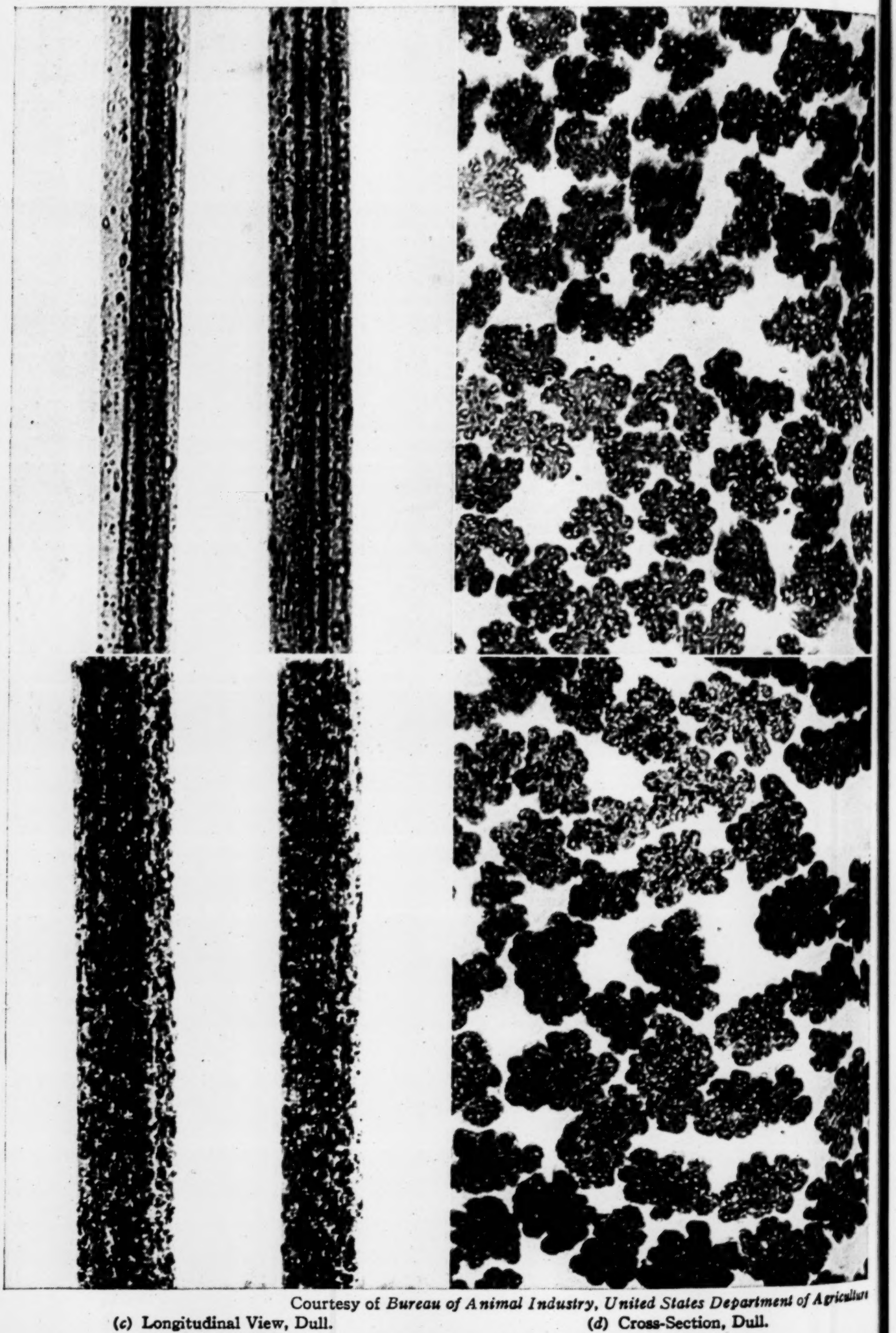


Courtesy of *Bureau of Animal Industry, United States Department of Agriculture*

(a) Longitudinal View. **(b) Cross-Section.**

FIG. 1.—Viscose Rayon Fibers, Bright (× 500).

(*a*) Longitudinal View, Semidull. **(*b*) Cross-Section, Semidull.**

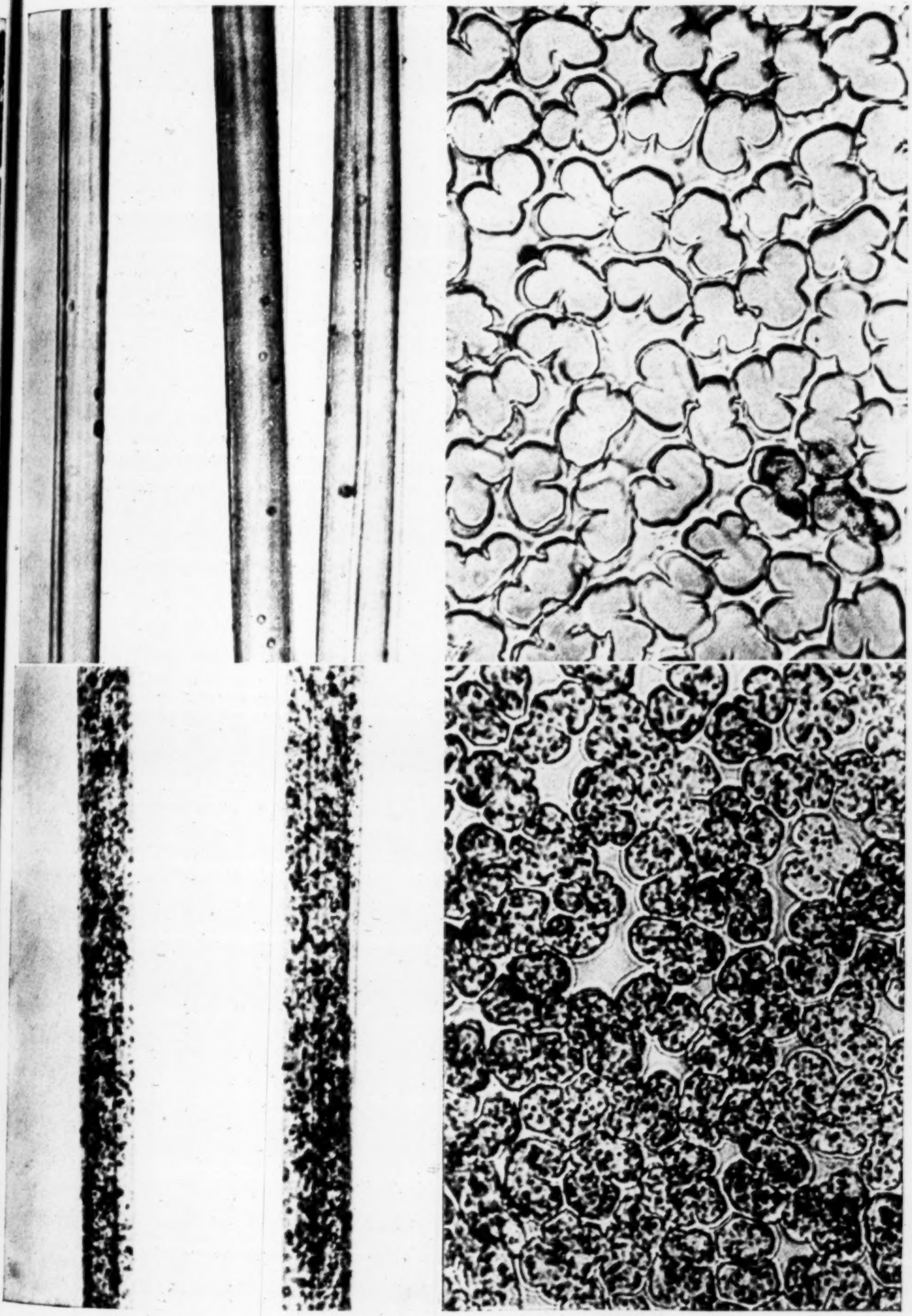


Courtesy of *Bureau of Animal Industry, United States Department of Agricultur*

(*c*) Longitudinal View, Dull. **(*d*) Cross-Section, Dull.**

FIG. 2.—Viscose Rayon Fibers (× 500).

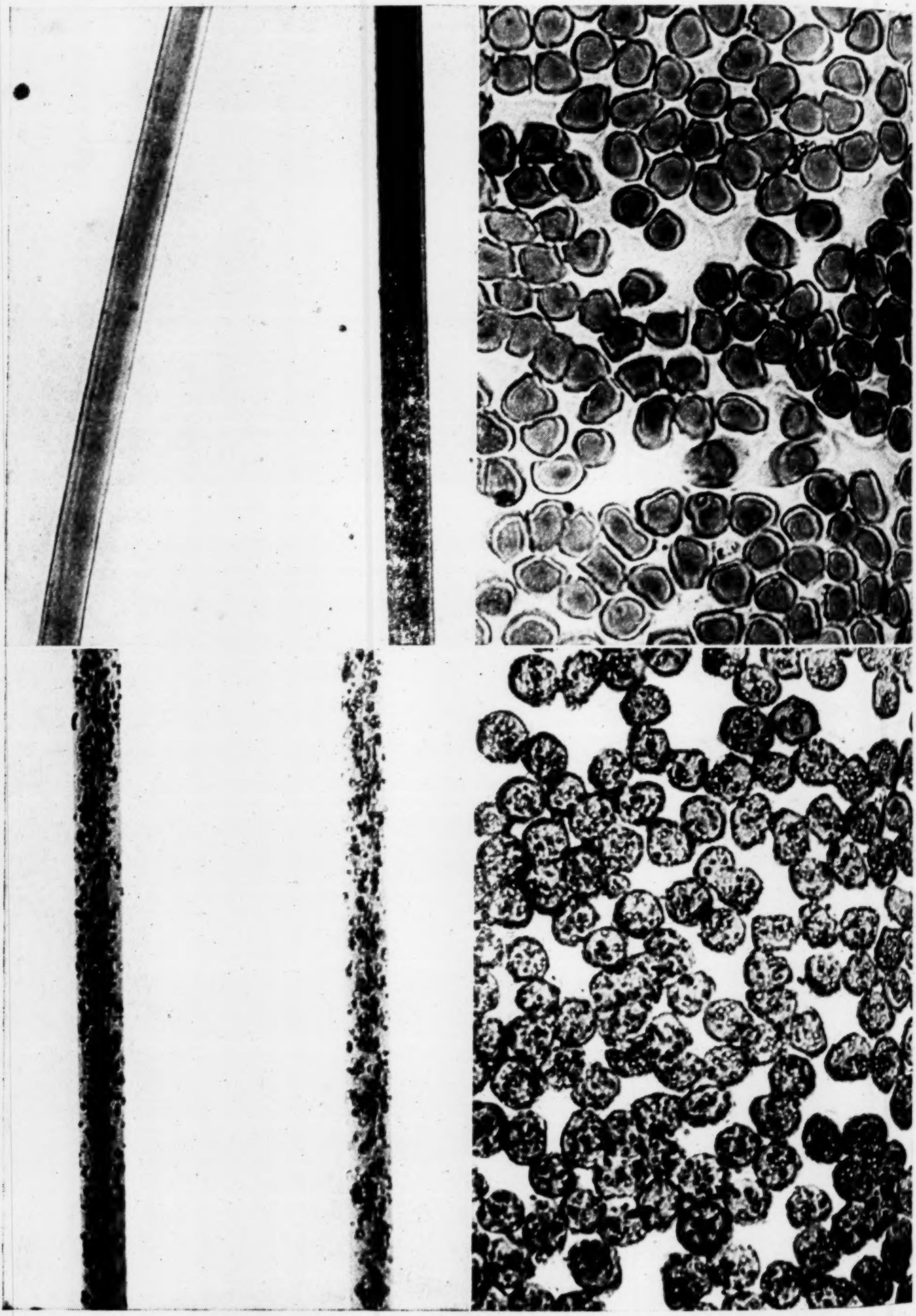
(*a*) Longitudinal View, Bright. (*b*) Cross-Section, Bright.



Courtesy of *Bureau of Animal Industry, United States Department of Agriculture*

(*c*) Longitudinal View, Dull. (*d*) Cross-Section, Dull.

FIG. 3.—Acetate Rayon Fibers (× 500).



(*a*) Longitudinal View, Regular Luster.

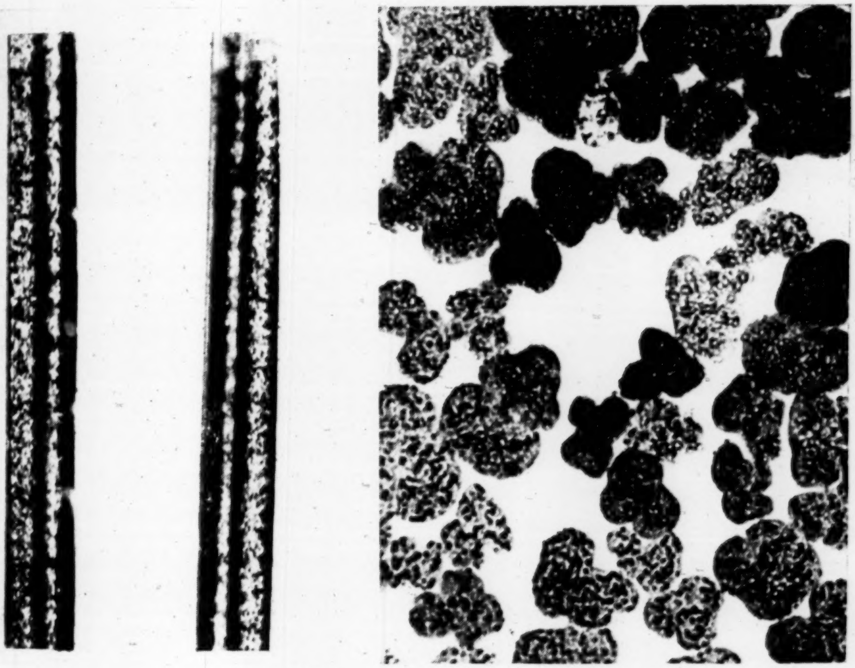
(*b*) Cross-Section. Regular Luster.

Courtesy of *Bureau of Animal Industry, United States Department of Agricultur*

(*c*) Longitudinal View, Semidull.

(*d*) Cross-Section, Semidull.

FIG. 4.—Cuprammonium Rayon Fibers (× 500).



Courtesy of *Bureau of Animal Industry, United States Department of Agriculture*

(*a*) Longitudinal View. (*b*) Cross-Section.

FIG. 5.—Nitrocellulose Rayon Fibers, Dull (× 500).

TENTATIVE SPECIFICATIONS AND METHODS OF TEST

FOR

FINENESS OF WOOL TOPS[1]

A.S.T.M. Designation: D 472 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.[2]

Scope

1. These specifications and methods of test are applicable to the grading of wool tops for fineness.

TABLE I.—REQUIREMENTS FOR WOOL TOPS.

Grade		80's	70's	64's	60's	58's	56's	50's
Fineness Range, microns	Min	18.5	20	21.5	24.5	26	27.5	31
	Max	19.5	21	22.5	25.5	27	29	33
Fine fibers, min., per cent	Group A....10 to 20 microns	60	50	36	18	16	9	4
	Group B....10 to 25 microns	92	84	....	....	....	....	....
	Group C....10 to 30 microns	....	....	94	83	74	64	45
Coarse fibers, max., per cent	Group W....25 to 30 microns	8	14	....	....	....	....	....
	Group X....30 to 40 microns	....	2	6	17	24	31	45
	Group Y....40 to 50 microns	....	....	....	....	2	5	10
Permissible variation for fibers coarser than specified		0.25	0.25	0.25	0.5	0.75	0.75	1.0
Minimum number of fibers required for test, per sample		400	400	600	800	800	1200	1200

Definition

2. *Wool Top.*—Wool top is a continuous untwisted band of wool fibers from which the shorter fibers or noils have been removed by combing.

NOTE.—Commercial wool tops usually range from 4 to 8.5 oz. per 10 yd. and are wound in balls ranging from 5 to 15 lb. in weight.

SPECIFICATIONS

Fineness

3. The average fiber width, the minimum percentage of fine fibers and the maximum percentage of coarse fibers in wool top, as determined by test, shall conform to the requirements specified in Table I.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

[2] Accepted for publication as tentative by Committee E-10 on Standards, August 26, 1937.

METHODS OF TESTING

Sampling

4. Samples of at least one yard in length shall be submitted in accordance with the following requirements:

SIZE OF LOT, LB.	NUMBER OF BALLS SAMPLED
Under 5000	2
5000 to 20,000	3
Over 20,000	3 for each 20,000 lb.

Minimum Number of Fibers for Test

5. The minimum number of fibers required for each determination shall be the number per sample as prescribed in Table I for the specific grade times the number of samples submitted (Section 4). The minimum number of fibers shall be measured in groups of 100 fibers for each test specimen (Sections 8 and 14). The test specimens shall be taken from points uniformly distributed along the length of the sample.

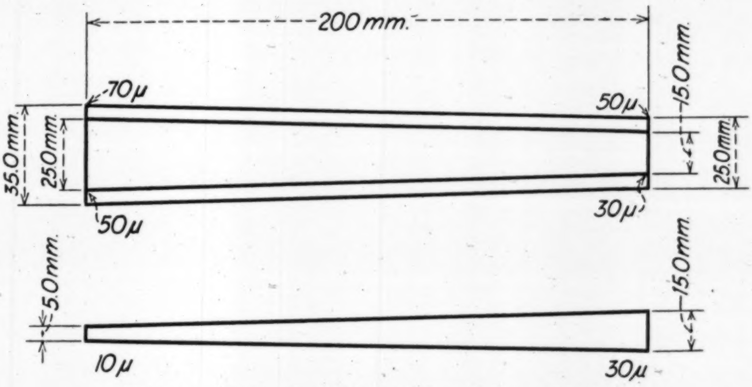


FIG. 1.—Wedge Ruler Consisting of Three Wedges.

Fineness Test

6. The fineness of wool tops shall be determined in accordance with either the width method or the cross-section method.

WIDTH METHOD

7. The width method is based on the projection of the image of the fiber at a high magnification through a microscope on a wedge ruler. The width of the image of the fiber is recorded on the wedge ruler by which the fibers are automatically sorted according to their width.

Apparatus

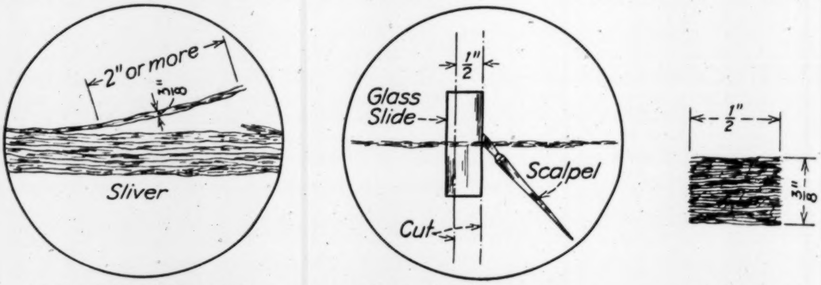
8. (*a*) *Microscope*.—The microscope shall have a movable stage and fixed body tube, an 8-mm. objective and a 12.5× eyepiece to give a magnification of 500 diameters, and shall be built for projection.

(*b*) *Wedge Ruler.*—The wedge ruler shall consist of a sheet of white cardboard on which is printed three wedges of the dimensions shown in Fig. 1.

(*c*) The portion of the projected field within which measurements are to be made shall not be larger than a circle 4 in. in diameter and shall be centrally located in the projected field.

Test Specimen

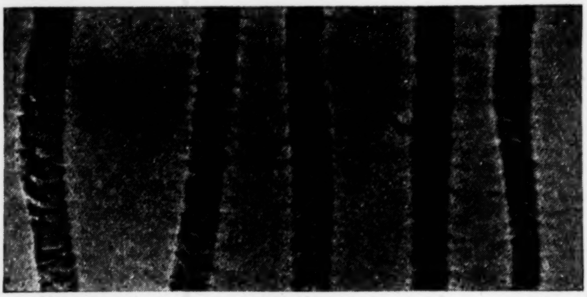
9. Test specimens, each containing about 120 fibers shall be prepared in the following manner: From the top of the sample separate a strand of fibers approximately ⅛ in. in diameter and 2 in. or more in length, Fig. 2 (*a*). From this strand cut with a razor blade or a pair of scissors, a small section approximately ½ in. in length, Fig. 2 (*b*) and (*c*). Place a drop of glycerine on a glass slide and mount the fiber bundle. Spread the fibers uniformly in the glycerine with the aid of dissecting needles, and then cover the fibers thus mounted with a cover glass. The final arrangement of the fibers on the slide should be similar to those shown in Fig. 2 (*d*).



(*a*) Separation of Strand of Fibers from Sample.

(*b*) Cutting ½-in. Section.

(*c*) Cut Sample.



(*d*) Arrangement of Fibers on Slide.

FIG. 2.—Method of Preparing Test Specimen.

Measurement of Width

10. The width of the fibers shall be determined by the following procedure: Move the slide by means of the mechanical stage until the extreme

left- or right-hand edge of the cover glass is projected in the measurement circle. Disregarding the first few fibers, bring the image of a fiber into sharp focus on the wedge ruler. Turn the wedge ruler until the image of the fiber to be measured is projected directly between the two lines of the wedge. Mark on the wedge ruler by a fine line, Fig. 3, the place where the wedge has exactly the same width as the image of the fiber. The image of a fiber which is not uniform in width shall be measured either at a point which appears to be the mean value between the greatest and least width (*a* in Fig. 4), or at the points of greatest and least width (*b* and *c* in Fig. 4), and averaging the two readings to determine the mean width. Bring the image of the next fiber into focus and measure as directed above and repeat the measurements on successive fibers until 100 fibers have

TABLE II.—EXAMPLE OF CALCULATIONS FOR FINENESS.[1]

CELL	CELL MIDPOINT	CELL BOUNDARY	DEVIATION IN CELLS FROM A, x	OBSERVED FREQUENCY, y	yx	yx^2
		10.0				
No. 5	11.25		..	..	..	..
		12.5				
No. 6	13.75		0	3	..	..
		15.0				
No. 7	16.25		1	17	17	17
		17.5				
No. 8	18.75		2	23	46	92
		20.0				
No. 9	21.25		3	21	63	189
		22.5				
No. 10	23.75		4	14	56	224
		25.0				
No. 11	26.25		5	10	50	250
		27.5				
No. 12	28.75		6	10	60	360
		30.0				
No. 13	31.25		7	2	14	98
		32.5				
				$\Sigma y = 100$	$\Sigma yx = 306$	$\Sigma yx^2 = 1230$

A = Midpoint of Cell No. 0 = 13.75 microns

m = Number of units per cell = 2.5 microns

$n = \Sigma y$ = Number of observations = 100

$$a = \frac{\Sigma yx}{\Sigma y} = \frac{306}{100} = 3.06 \qquad c = a^2 = (3.06)^2 = 9.36$$

$$b = \frac{\Sigma yx^2}{\Sigma y} = \frac{1230}{100} = 12.30 \qquad d = b - c = 12.30 - 9.36 = 2.94$$

Arithmetic Mean Width, $\overline{X} = A + (m \times a) = 13.75 + (2.5 \times 3.06) = 21.40$

Standard Deviation, $\sigma = m\sqrt{d} = 2.5 \times 1.72 = 4.30$

$$\left.\begin{array}{r}\text{Limits for } \overline{X} \\ (P_s = 0.90 \text{ (Chances, 9 in 10))}\end{array}\right\} = \pm \frac{1.645\sigma}{\sqrt{n-3}} = \pm 0.72$$

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[1] Based on A.S.T.M. Manual on Presentation of Data, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 453 (1933).

been measured. Repeat the above procedure on additional test specimens until the required number of fibers per sample as specified in Table I have been measured.

Calculations

11. The fineness of wool top shall be expressed in terms of the arithmetical mean width of the specified minimum number of fibers in microns and frequency. The dispersion factor of a wool top grade may be expressed in terms of the standard deviation and the limits for the average width may be expressed in terms of $P_s = 0.90$, which defines the ranges within which universe averages will fall 9 times in 10. (An example of the calculations for fineness is shown in Table II.).

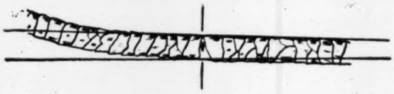

FIG. 3.—Marking Wedge Ruler Where Fiber is of Uniform Diameter.

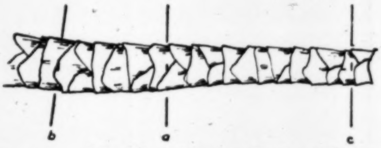

FIG. 4.—Marking Wedge Ruler Where Fiber is not of Uniform Diameter.

CROSS-SECTION METHOD

12. (*a*) In the cross-section method, the image of the cross-section of the fibers is projected through a microscope upon sensitized paper, at a magnification of 500×. The sensitized paper is then developed, fixed and dried. The images of the fibers on the paper are measured in two directions at right angles to each other by means of a celluloid wedge measure, graduated to read directly in microns, or by means of a bidiameter scale.

(*b*) For direct measurement the image of the cross-section of the fibers may be projected on a sheet of white paper and the images of the fibers measured in accordance with Paragraph (*a*).

Apparatus

13. (*a*) *Microscope.*—Laboratory and dissecting microscopes and a projection microscope as specified in Section 8 (*a*) will be required.

(*b*) *Cross-Section Device.*—The cross-section device illustrated in Fig. 5 shall be used in making the cross-section of the fibers. It consists of two parts—the fiber holder, *C*, with the fiber slot, and the slide holder, *D*, in which is rigidly held a flat surfaced metal plunger, with right angle flanges to fit in the slot of the fiber holder (sliding fit.). When assembled, the shape of the cross-section device is as that of a microscopic slide, 1 in. in width and 3 in. in length. The guides on each side of the slide holder keep the fiber holder steady and help hold the fiber and slide holder together, as shown in the side view, B, Fig. 5.

(*c*) *Wedge Measure.*—The wedge measure illustrated in Fig. 6 shall be made from celluloid 0.0020 in. in thickness.

(*d*) *Bidiameter Scale.*—The bidiameter scale, Fig. 7, shall be made on a 2 by 2½-in. piece of glass. The scale rulings shall cover an area of 3 by 4 cm. Each small division of 1 mm. is equal to 2 microns at a magnification of 500 diameters. The surface upon which the scale rulings appear must be protected from being scratched by affixing a narrow strip of celluloid on each of the 2-in. edges of the scale.

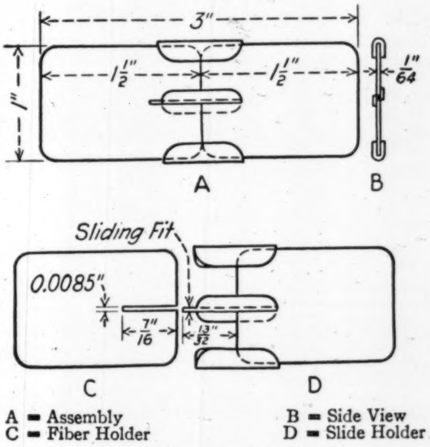

FIG. 5.—Cross-Section Device.

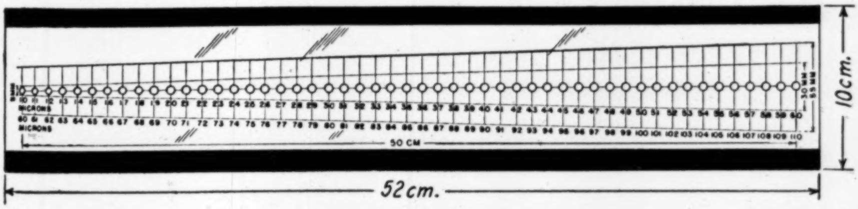

FIG. 6.—Wedge Measure.

Test Specimen

14. Test specimens shall be prepared in the following manner: Separate a strand of about 150 fibers from the sample (Fig. 2(*a*)), care being taken not to disturb the distribution of the fibers as they occur in the sample. Insert this strand of fibers in the fiber holder and push the slide of the slide holder into the fiber slot until the fibers are held securely in place. The slide of the slide holder should have just sufficient tension upon the fibers to hold them without distorting their shape. Cut the fibers off close on both sides of the fiber holder with scissors. Apply a

drop of celluloid solution on one side and allow to dry. Then cut the fibers off flush on both sides of the fiber holder by means of a sharp razor blade, the edge of which shall appear smooth and free from nicks when examined under the microscope at a magnification of 100 diameters. When cutting the fibers, keep the bevel of the razor blade parallel to the surface of the fiber holder. This may best be accomplished by using a binocular microscope, magnifying about 12 diameters. After the cross-section is made, examine it under a microscope at a magnification of about 300 diameters. When the fibers are all smoothly cut and each fiber is clearly defined, the cross-section is ready for projection.

Projecting Cross-Section of Specimen

15. With the projection microscope set for a magnification of 500 diameters, the cross-section device shall be placed on the stage of the microscope and the image of the cross-section of the specimen shall be focused through an orange color filter upon a sheet of white paper. The illumination shall be uniform for different cross-sections. To secure this condition a filter of the proper depth of color should be used according to the intensity of the light passing through the cross-section.

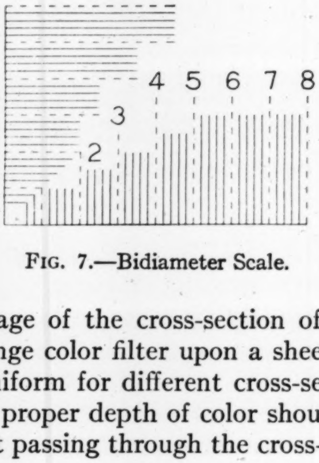

FIG. 7.—Bidiameter Scale.

Measuring Fibers for Fineness

16. (*a*) The cross-section of the fibers (Section 12 (*a*) or (*b*)) shall be measured in microns for their greatest and least diameters, at right angles to each other. The first few fibers near any edge of the cross-section device shall not be measured. Measurement of successive fibers, without skipping, shall be made in accordance with Paragraphs (*b*) or (*c*) until 100 fibers have been measured.

(*b*) By means of the celluloid wedge measure (Fig. 6) the cross-sections of the fibers shall be measured in microns for their greatest and least diameters, at right angles to each other. As each fiber is measured, it shall be numbered through the hole in the wedge.

(*c*) By means of the bidiameter scale (Fig. 7) the greatest and least diameters of the cross-section of a fiber shall be measured as follows: Place the extreme left vertical line of the scale tangent to the periphery of the fiber cross-section at a point of major diameter. Adjust the long horizontal base line tangent to the periphery at a point of minor diameter. With the scale in this position both diameters of the fiber can be read directly in microns with one setting. A reading can easily be made to one-half of a division, which is equivalent to one micron.

Calculations

17. The fineness of each fiber shall be obtained by averaging its greatest and least diameters. The fineness of wool tops shall be expressed in terms of the arithmetical mean diameter of the specified minimum number of fibers in microns and frequency. Other statistical values may be obtained as set forth in Section 11.

TENTATIVE METHODS OF TESTING WOOL FELT[1]

A.S.T.M. Designation: D 461 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These methods of test are applicable to fabrics which are neither woven nor knitted, but built up by the interlocking of wool fibers by mechanical work, chemical action, moisture, and heat. Felt may be produced by one or a combination of any of these factors. It may consist of wool, or wool mixed with other fibers, or fibers other than wool.

Standard Condition

2. (*a*) Standard condition of a felt shall be that reached by the fabric in moisture equilibrium in an atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of ±2 per cent is permitted in relative humidity and ±2 F. (1.1 C.) in temperature.

(*b*) *Moisture Equilibrium.*—It shall be considered that moisture equilibrium is reached when two successive weighings not less than 30 min. apart show not more than 1.0 per cent change in weight.

Test Condition

3. All felts shall be tested under standard conditions as described in Section 2.

METHODS OF PHYSICAL TESTING

Length

4. The length of a roll or piece shall be determined by running the felt over a measuring drum of known circumference, from which the yardage is registered by a dial or counter driven by a chain, or other positive non-slip mechanism which measures the length of the back of the felt and is independent of its thickness. Just enough uniform tension shall be used to keep the felt running flat and true. As an optional method, measurement may be made with a steel tape, care being taken to insure that the material lies flat without excess tension.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

Width

5. The width of a roll or piece shall be determined at five different places uniformly distributed along the full length of the roll or piece, and may be determined at the same time as the length.

Thickness

6. (*a*) *Gage.*—The thickness of pieces or sheets shall be measured by a dial micrometer, consisting of an anvil for supporting the specimen, a circular presser foot acting under a dead-weight load, and a dial graduated to read directly in thousandths of an inch.

(*b*) *Supporting Anvil.*—The anvil for supporting the specimen shall have a flat horizontal surface of minimum linear diameter of 2 in. In measuring the thickness of large pieces or sheets of felt, the supporting anvil shall be in the same plane as the supporting table.

(*c*) *Presser Foot.*—The presser foot shall have an area of 1 sq. in. ±0.0025 sq. in. (1.129 in. in diameter). It shall be sufficiently thick to insure rigidity and the edges shall be rounded off with a radius of 0.016 in. ±0.001 in. The surface of the presser foot shall be parallel to that of the supporting anvil.

(*d*) *Load.*—The presser foot shall be actuated by a freely acting total dead-weight load of 10 oz. ±0.5 oz. Less than 0.5 oz. shall be required to overcome friction and to produce perceptible motion of the presser foot when counterbalanced. The load shall be applied gradually and without shock. The thickness reading shall be taken at least 10 sec. after the load is applied.

(*e*) The thickness of cut parts[1] having a minimum linear dimension greater than 1 in. shall be measured by the dial micrometer as prescribed in Paragraphs (*a*) to (*d*).

Weight

7. A representative specimen containing not less than 40 sq. in. shall be cut from the felt. The specimen should be taken from midway between the outer edges. No specimen shall be taken within 6 in. of either side or end. The specimen shall be conditioned (Section 2) and then weighed on a balance sensitive to 0.1 per cent of the total weight. The weight shall be expressed in ounces per square yard.

Breaking Strength

8. (*a*) *Testing Machine.*—The breaking strength test shall be made on an approved type of testing machine of proper capacity such as described in the Standard Specifications for Textile Testing Machines (A.S.T.M. Designation: D 76) of the American Society for Testing Materials.[2] The

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[1] See Editorial Note, p. 1230.

[2] 1937 Supplement to Book of A. S. T. M. Standards, p. 241.

jaws shall not be less than 2 in. in width, and shall be set 3 in. apart. The speed of the pulling jaw shall be $12 \pm \frac{1}{2}$ in. per min.

(*b*) *Test Specimens.*—Specimens for the breaking strength test shall be 2 by 10 in. Eight test specimens, four taken lengthwise and four crosswise of the piece, shall be cut with a die in such a manner that the 10-in. dimension will be parallel to or perpendicular to the length direction, respectively. All specimens shall be conditioned in accordance with Section 2 before testing.

(*c*) *Procedure.*—The breaking strength of each conditioned specimen of felt shall be determined and the average strength in the length and width directions shall be reported separately in pounds per 2-in. width.

Splitting Resistance

9. (*a*) *Testing Machine.*—The splitting resistance shall be determined on a testing machine conforming to the requirements prescribed in Section 8 (*a*).

(*b*) *Test Specimens.*—Specimens for the splitting resistance test shall be 2 by 10 in. Eight test specimens, four taken lengthwise and four crosswise of the piece, shall be cut with a die in such a manner that the 10-in. dimension will be parallel to or perpendicular to the length direction, respectively. All specimens shall be conditioned in accordance with Section 2 before testing.

(*c*) *Procedure.*—The conditioned specimen shall be split in thickness by cutting with a knife within the middle third of the thickness for a distance of approximately 2 in. from one end only. One of these lips shall be clamped in each jaw of the testing machine, the jaws being brought close enough together for the purpose. The machine shall then be started and the average load in pounds necessary to pull the two sections of felt wholly apart shall be recorded as the splitting resistance in pounds. The test results on the four specimens cut lengthwise and the four cut crosswise shall be respectively averaged and reported separately as the splitting resistance in pounds per 2-in. width.

NOTE.—This test is not applicable to felts where the thickness is less than $\frac{3}{16}$ in. For material less than $\frac{3}{16}$ in. in thickness breaking strength only is recommended as an indicative test.

METHODS OF CHEMICAL ANALYSIS

Matter Soluble in Carbon Tetrachloride

10. (*a*) *Sample.*—A sample of approximately 8 g. of the felt shall be prepared for chemical analysis by cutting into pieces approximately $\frac{1}{8}$-in. square.

(*b*) Approximately 5 g. of the material prepared in accordance with Paragraph (*a*) shall be dried at 105 to 110 C. to constant weight. This shall be designated as the *original oven-dried specimen* and its weight as weight *B*. The original oven-dried specimen shall be extracted with carbon tetrachloride in a Soxhlet apparatus for 4 hr. The carbon tetrachloride shall then be evaporated from the extract and the residue dried at 105 to 110 C. to constant weight. This residue is matter soluble in carbon tetrachloride and shall be reported as a percentage of the weight, *B*, of the original oven-dried specimen.

Matter Soluble in Water

11. The fibrous residue from the carbon tetrachloride treatment (Section 10 (*b*)) shall be dried at 105 to 110 C. to constant weight. It shall then be covered with distilled water and boiled for 15 min. The solution shall be decanted through a filter into a weighed evaporating dish. The boiling with distilled water and decanting shall be repeated four times. The combined solutions shall be evaporated and the residue dried at 105 to 110 C. to constant weight. This residue is matter soluble in water and shall be reported as a percentage of the weight, *B*, of the original oven-dried specimen (Section 10 (*b*)).

Wool Determination

12. In this procedure wool is determined by chemical analysis and not on a *fiber basis*. The fibrous residue from the water extraction (Section 11) shall be dried at 105 to 110 C. to constant weight. This gives the weight of the moisture-free fibrous residue, weight *C*. The moisture-free fibrous residue shall be placed in an Erlenmeyer flask and covered with 105 to 150 ml. of sodium hydroxide solution (5 per cent). A reflux condenser shall then be attached and the solution boiled for 20 min. The undissolved fibers shall be collected on a Gooch filter or a 100 mesh wire screen and washed with distilled water until neutral to litmus paper. The residue shall then be dried at 105 to 110 C. to constant weight. This gives the weight of the vegetable matter, weight *D*. The wool content shall be calculated as a percentage of the weight of the original oven-dried specimen from the following formula:

$$\text{Wool (dry basis), per cent} = \frac{100}{B}\left(C - \frac{D}{0.95}\right)$$

where B = the weight of the original oven-dried specimen (Section 10 (*b*)),
C = the weight of the moisture-free fibrous residue from the water extraction, and
D = the weight of the washed and dried residue (vegetable matter).

NOTE 1.—The above formula is based upon the assumption that 5 per cent of the cotton is dissolved by a sodium hydroxide solution (5 per cent).

NOTE 2.—The wool content may include hair and other animal fibers similar to wool which by hydrolysis are removed with the wool. For detection of substitutes, see Section 14.

Ash

13. The balance (approximately 3 g.) of the material prepared in accordance with Section 10 (*a*) shall be placed in an oven and dried at 105 to 110 C. to constant weight, and allowed to cool in a desiccator. A 2-g. portion of the dried specimen shall be weighed quickly in a tared crucible. The crucible shall be placed in a muffle furnace and the temperature shall be raised slowly to 800 to 1000 C., care being taken to avoid loss of ash by quick ignition or by subjecting the specimen to strong drafts. When all combustible matter has been volatilized, the crucible shall be placed in a desiccator, cooled, and weighed. The ignition and subsequent cooling in a desiccator shall be repeated until a constant weight is obtained. The ash shall be reported as a percentage of the weight of the oven-dried material.

Microscopic Examination

14. In the determination of wool content (Section 12), all animal fibers are included as wool. If it is desired to detect the presence of, and identify, fibers other than wool (such as other animal fibers, vegetable and synthetic fibers), the felt shall be subjected to a microscopical examination such as is described in Tentative Methods for the Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles (A.S.T.M. Designation: D 276 – 37 T) of the American Society for Testing Materials.[1]

EDITORIAL NOTE

Methods for determining the thickness of small cut parts are being studied by the committee by means of cooperative interlaboratory tests and when formulated will be incorporated in Section 6. The following description of these methods is accordingly published herewith as information only:

(*a*) The thickness of small cut parts may be measured by the dial micrometer by cutting to fit and closely nesting together a sufficient number of pieces to give a minimum linear dimension of 1.25 in.

(*b*) The thickness of cut parts having a minimum linear dimension less than 1 in. and purchased under a specification which includes dimensions, weight per square yard, and permissible tolerances, may be determined by weighing a number of parts and calculating the thickness in terms of the specified weight per square yard.

Example.—A cut part of annular shape of the following dimensions, 1.00 in. in outside diameter, 0.75 in. in inside diameter, 0.25 in. in thickness, will have an area of 0.3436 sq. in., and if cut from a ¼-in. felt weighing 4 lb. per sq. yd., 100 pieces will weigh 0.106 lb. If 100 pieces selected at random from a shipment actually weigh 0.092 lb., the thickness would be reported as 0.217 in.

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[1] See p. 1174.

TENTATIVE METHODS OF TESTING AND TOLERANCES

FOR

CERTAIN WOOL AND PART WOOL FABRICS[1]

A.S.T.M. Designation: D 462 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These methods of testing and tolerances apply to fabrics made wholly or in part of wool yarn, excluding blankets, pile fabrics, knitted fabrics, and wool felt.

Classification[2]

2. The terms "100 per cent," "All," "Pure," and similar expressions, when used to modify or in connection with wool, woolen, or worsted fabrics, are synonymous and shall be used only in referring to fabrics, the fiber content of which shall be 100 per cent wool.

3. The terms "Wool," "Woolen," or "Worsted," with the expressions "decorated with silk," "with mercerized decorations," and similar expressions, shall be used only in referring to fabrics of which the wool fiber content is not less than 95 per cent by weight of the total fiber content.

4. The terms "Wool," "Woolen," or "Worsted," when used in referring to fabrics of which the wool fiber content is less than 95 per cent by weight of the total fiber content, shall be preceded by a figure indicating the actual minimum percentage of wool fiber present.

TOLERANCES

Tolerances

5. Tolerances are the limits within which the specified characteristics of the fabric must come to constitute a good delivery on contract.

Wool Content

6. The wool content shall not be more than 2 per cent under the required content as specified in Sections 2 and 3.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

[2] This classification and the methods of chemical analysis in these Tentative Methods conform in substance, but not in form, with the Recommended Commercial Standard for Wool and Part Wool Fabrics (TS-2300) promulgated by the U. S. Department of Commerce.

Count

7. The number of threads per inch, in both the warp and the filling, shall be not more than 3.5 per cent over or under the specified count.

Breaking Strength

8. The breaking strength shall be not less than that specified.

METHODS OF PHYSICAL TESTING

Methods

9. The methods of testing described in the Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39) [1] or in the Tentative Methods for the Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles (A.S.T.M. Designation: D 276 - 37 T)[2] of the American Society for Testing Materials shall be used where applicable to wool and part wool fabrics. These instances are indicated below by references to the specific sections of Methods D 39 or D 276 which are applicable.

Width

10. The width of the fabric shall be determined in accordance with Section 4 of Standard Methods D 39. The measurements shall be made inside the selvages and to an accuracy of $\frac{1}{16}$ in.

Weight

11. The weight of the fabric shall be determined in accordance with Section 6 of Standard Methods D 39.

Count

12. The number of threads per inch, both warp and filling, shall be determined in accordance with Section 7 of Standard Methods D 39.

Breaking Strength

13. The breaking strength of the fabric shall be determined by the grab method in accordance with Section 10 of Standard Methods D 39.

Mechanical Analysis

14. Where practicable, the different kinds of fiber in a fabric may be separated mechanically from each other after removing the sizing, finishing, and non-fibrous materials in accordance with Section 15.

METHODS OF CHEMICAL ANALYSIS

Total Sizing, Finishing and Other Non-Fibrous Materials

15. The total sizing, finishing and other non-fibrous materials shall

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[1] 1936 Book of A.S.T.M. Standards, Part II, p. 1334.

[2] See p. 1174.

be determined in accordance with Section 3 of Tentative Methods D 276.

Chemical Tests for Determining Wool Content

16. If microscopic examination indicates no other fibers present but wool, cotton, silk, or rayon, the material shall be tested in accordance with the methods referred to in the following Paragraphs (*a*) to (*c*):

(*a*) *Mixtures of Wool with Cotton, or Rayon, or Both.*—The wool content shall be determined by the sulfuric acid method in accordance with Sections 9 and 10 of Tentative Methods D 276.

(*b*) *Mixtures of Wool and Silk.*—The wool content shall be determined from the residue after the removal of the silk in accordance with Section 5 of Tentative Methods D 276.

(*c*) *Mixtures of Wool, Cotton, Rayon, Acetate, and Silk.*—The wool content shall be determined in accordance with Sections 4 to 10 of Tentative Methods D 276.

Shrinkage in Sponging[1]

17. (*a*) *Test Specimen.*—The specimen of cloth selected for test shall be at least 20 in. square. The specimen shall be laid out without tension on a flat surface, care being taken that the cloth is smooth and free from wrinkles or creases. Three distances, each of 18 in. (or longer when the size of the specimen permits), shall be marked off on the specimen in both the warp and filling directions. The distances shall be parallel to the warp or filling threads, at least 6 in. apart, and at least 1 in. from all edges of the specimen. Satisfactory marks may be made with indelible ink and fine-pointed pen or by sewing fine threads into the cloth.

(*b*) *Wetting.*—The specimens shall be immersed in and saturated with water at a temperature of 75 to 85 F. (24 to 29 C.).

(*c*) *Drying.*—The specimens shall be removed from the water and wrapped in a clean white towel or similar cloth. The excess water shall be removed from them in a centrifuge or by squeezing by hand. Wringing by hand or with squeeze rolls is not permissible as either may distort the cloth. The specimens shall then be spread out on a horizontal screen or similar perforated surface and dried in a current of air. If a heated drying chamber is not available, the specimens may be dried at room temperature in a current of air from an electric fan. The dried specimens shall then be dampened with water and allowed to stand for 5 min.

(*d*) *Pressing.*—The specimens shall be pressed either with a flat-bed press or a hand iron, care being taken in handling them to avoid any strain on the cloth. Each specimen shall be smoothed to remove wrinkles before pressing. When a hand iron is used, the iron shall not be slid back and forth on the specimen but simply pressed down upon it in a manner simulating the action of a flat-bed press. The specimen shall be allowed to cool before it is measured.

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[1] This shrinkage in sponging test conforms in substance, but not in form, with Section XV of the Federal Specification for Textiles; Test Methods (CCC-T-191).

(*e*) *Results.*—The pressed specimen shall be laid out without tension on a flat surface, care being taken that the cloth is smooth and free from wrinkles or creases, and the distances which were marked off shall be measured again. The shrinkage in the warp and in the filling directions of cloth shall be calculated separately as the average obtained from the three marked distances. The shrinkage in sponging is the decrease in the distance measured and shall be expressed either as a percentage of the dimension before sponging or in inches per yard.

TENTATIVE METHODS OF TESTING PILE FLOOR COVERING[1]

A.S.T.M. Designation: D 418 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1935; Revised, 1936, 1937.

Scope

1. These methods of testing are intended to be applicable for testing pile floor coverings.

Sampling

Note.—The size of dissection samples and methods of sampling will be covered in later revisions of these methods.

Condition

2. (*a*) *Unconditioned.*—The weight of the fabric when samples for weight determination are cut from it is the unconditioned weight. A specification of the weight of a fabric must also specify the moisture condition.

(*b*) *Oven-Dry Condition.*—The oven-dry condition of pile fabrics is that state of dryness obtained on equilibrium of the material in a ventilated drying oven maintained at a temperature of 220 to 230 F. (105 to 110 C.).

(*c*) *Standard Condition.*—Standard condition of pile fabric is that reached by the fabric when in moisture equilibrium with a standard atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of ± 2 per cent is permitted in relative humidity and plus 10 F. (6 C.) in temperature.

(*d*) *Moisture Equilibrium.*—It shall be considered that moisture equilibrium is reached when, after free exposure to the standard atmosphere in motion, two successive weighings not less than 30 min. apart show not more than 1.0 per cent change in weight. Equilibrium shall be approached from the dry side (not oven dry).

Test Condition

3. All fabrics shall be tested under standard conditions as described in Section 2 (*c*).

Quantity

Length

4. The length of a roll or piece shall be determined by running the fabric over a measuring drum of known circumference, from which the

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

yardage is registered by a dial or counter driven by a chain, or other positive non-slip mechanism. This yardage shall be the length of the fabric and shall be independent of any effect of its thickness.

Width

5. (*a*) The width of a roll or piece shall be determined at five different places uniformly distributed along the full length of the roll or piece, and may be determined at the same time as the total length.

(*b*) Measurements shall be made to the nearest $\frac{1}{16}$ in.

(*c*) The average of the five measurements shall be the width.

Weight

6. (*a*) *Unconditioned Weight.*—Samples in a condition which is the same as the fabric they represent shall be rapidly weighed on a balance sensitive to 0.1 per cent of the total weight. The average of these weights shall be the unconditioned weight of the samples.

(*b*) *Conditioned Weight.*—The samples shall be allowed to come to standard condition as described in Section 2 (*c*). The average of the samples weighed under standard conditions shall be the conditioned weight of the samples and shall be used to calculate the conditioned weight of the fabric.

(*c*) *Oven-Dry Weight.*—One of the samples shall then be converted to the oven-dry condition as described in Section 2 (*b*). This shall be the oven-dry weight of the sample.

Moisture Content and Regain

7. (*a*) *Weight of Moisture.*—The difference between the unconditioned weight (Section 6 (*a*)) and the oven-dry weight (Section 6 (*c*)) shall be the weight of moisture present.

(*b*) *Moisture Content.*—The weight of moisture when calculated as a percentage of the unconditioned weight shall be the "moisture content" of the fabric.

(*c*) *Moisture Regain.*—The weight of moisture when calculated as a percentage of the oven-dry weight shall be the "moisture regain" of the fabric.

Total Thickness

8. Thickness of a fabric shall be determined by measuring to the nearest 0.001 in. the distance between the two plane surfaces of a fabric under a pressure of 0.100 ± 0.001 lb. per sq. in. distributed over a circular area 3.00 in. ± 0.01 in. in diameter. The pressure shall be applied slowly to avoid impact, and the fabric shall be protected from vibration during test. The average of five readings taken at different places, uniformly distributed over the area of the surface, shall be the thickness.

CONSTRUCTION

Back Thickness

9. The pile yarn shall be removed from a section of the fabric by clipping. A 5-in. diameter metal ring shall be seated on the clipped portion, a flame directed on the area within the ring, and the pile destroyed with alternate charring and brushing without damage to the back construction. The thickness of the remaining back construction shall be determined by measuring to the nearest 0.001 in. the distance between the two plane surfaces of the back construction under a pressure of 0.750 lb. ± 0.001 lb. per sq. in. distributed over a circular area 1.00 in. ± 0.01 in. in diameter. The pressure shall be applied slowly to avoid impact and the fabric shall be protected from vibration during test. The average of five readings taken at different places within the exposed back construction shall be the back thickness of the sample.

Net Pile Height

10. The net pile height is the difference between the total thickness and the back thickness, as determined in Sections 8 and 9.

Rows

11. The number of rows of tufts per inch shall be determined by counting a space of not less than 10 in. in at least three different places. No two count determinations shall include the same rows. The row count of the fabric shall be the average of the number of rows per inch in these three tests.

Pitch

12. The number of pile ends per inch of width shall be determined by counting the tufts in a space of not less than 10 in. in at least three different places staggered across the piece. The pitch of the fabric shall be the average of the number of pile ends per inch found in these three tests.

Shot

13. The number of shots in a single repeat of the weaving cycle, or the number of travels of filling yarn per row of tufts, shall be noted. This number shall be the shot of the fabric. In Axminster weaves or fabrics woven on a needle loom, the double thread or filling left by one movement of the needle shall be considered as one shot.

Dissection

14. Samples, in standard condition and which have not been oven-dried, shall be completely picked apart dividing the yarn according to its utility in the carpet, that is pile, warp, stuffer and filling. The debris from the dissection shall be included in the pile yarn weight.

Weights

15. (*a*) The yarns sorted according to their construction purpose shall be weighed under standard conditions.

(*b*) *Pile Yarn.*—The average of the weights of the samples of pile yarn shall be the conditioned weight of pile yarn.

(*c*) *Clean Pile Yarn.*—The average of the weights of the samples of pile yarn, after extraction to remove fats and after desizing to remove sizing materials, shall be the conditioned weight of clean pile yarn.

(*d*) *Warp Yarn.*—The average of the weights of the samples of warp yarn shall be the conditioned weight of warp yarn.

(*e*) *Stuffer Yarn.*—The average of the weights of the samples of stuffer yarn shall be the conditioned weight of stuffer yarn.

(*f*) *Filling Yarn.*—The average of the weights of the samples of filling yarn shall be the conditioned weight of filling yarn.

(*g*) *Weights per Square Yard.*—Each of the conditioned weights of pile, clean pile, warp, stuffer and filling yarn, determined in accordance with Paragraphs (*b*) to (*f*), shall be corrected to the weight per square yard by multiplying by the proper constant and shall be reported as the weight of pile yarn, clean pile yarn, warp yarn, stuffer yarn and filling yarn per square yard, respectively.

(*h*) *Clean Backing Yarns.*—When required, the average of the weights of the samples of warp yarn, stuffer yarn and filling yarn after extractions to remove fats and after desizing to remove size, shall be the conditioned weight of clean warp yarn, clean stuffer yarn and clean filling yarn, respectively.

NOTE.—Methods for desizing and fat removal will be developed and added later.

FASTNESS OF COLOR TO LIGHT

Standard

16. The standard for light fastness shall be a carpet having a wool content of approximately 27 oz. per sq. yd., a pile height of approximately 0.340 in., and made with yarn spun from white carpet wool, which has been stock dyed by the following procedure: The dye solution shall contain 2 per cent of Indigotine, conc. C. I. 1180 (DuPont), 10 per cent of calcined Glauber's salt and 5 per cent of sulfuric acid, based on the weight of the wool. For 1 part of wool by weight, 30 parts of the solution by weight shall be used. The wool shall be dyed in this solution at 90 C. for 45 min.

NOTE.—The fading characteristics of this standard are on record in the National Bureau of Standards in the form of spectrophotometric curves to assure accurate duplication of the standard when necessary. The standard may be obtained at cost from Alexander Smith and Sons Carpet Co., Yonkers, N. Y., by ordering sample ER–312 No. 1687.

Test Specimens

17. Test specimens for the determination of fastness to light shall be approximately 2½ by 4 in. and so cut that the 4-in. dimension shall be parallel to the direction of the chain or warp.

Apparatus

18. The apparatus for exposing the test specimens shall be a Fade-Ometer or similar machine by which fading is produced comparable to that by sunlight. The frame for holding the test specimens shall be so constructed as to permit the mounting of the specimen in such a manner that it cannot bend away from the front of the frame at any point, that the pile touches the front of the frame lightly, that the lower part of the specimen is exposed, and that the exposed area, approximately 1½ by 2 in., is completely surrounded by an unexposed area.

Procedure

19. Two test specimens prepared in accordance with Section 17 and two standards shall be mounted in the frame in such a manner that if there is a noticeable lay to the pile, one directional component of the lay of the pile shall point toward the bottom of the specimen. The four specimens shall then be placed in the Fade-Ometer at the same time. When the standards show a slight fading (about 20 hr.), one standard and one test specimen shall be removed and the number of hours of exposure noted. The exposure of the remaining standard and test specimen shall be continued until they have been exposed for twice the length of time required for the first fading.

Results

20. Each test specimen shall be compared with the standard against which it has been run. The fastness of the color to light shall be considered satisfactory if, after both the first and second periods of exposure, the change in color in the test specimen is less than that in the standard.

TENTATIVE METHOD OF TEST

FOR

VOLUMETRIC DETERMINATION OF SMALL AMOUNTS OF COPPER IN TEXTILES[1]

A.S.T.M. Designation: D 463 – 37 T

This is a Tentative Standard and under the Regulations of the Society is subject to **annual revision.** Suggestions for revision should be addressed to the Headquarters of the **Society,** 260 S. Broad St., Philadelphia, Pa.

Issued, 1933; Revised, 1936, 1937.

Scope

1. This method of test applies to all textile materials whose content of copper is less than 0.01 per cent but greater than 100 micrograms (1 microgram = 0.001 mg.).

Samples

2. The sample for analyses shall be as fairly representative of the material as possible. Each sample shall weigh (preferably) approximately 10 g. but in no case shall the weight be less than 5 g.

Blanks

3. Blank tests shall be run on all determinations and deductions made accordingly.

Number of Tests

4. A minimum of three determinations shall be made in every case and the average value taken as the true value. In cases of dispute an average of at least five determinations shall be taken.

Reagents

5. All reagents shall be of c.p. quality as prescribed by the American Chemical Society.

Special Solutions Required

6. (*a*) *Standard Copper Solution.*—Electrolytic copper foil of known purity shall be used for the preparation of the standard copper solution. The foil shall be cleaned by scraping down to the bright metal. Exactly 1 g. shall be dissolved in the least required amount of dilute HNO_3 (2:1, by volume) in an Erlenmeyer flask. Approximately 10 ml. of this diluted acid will be required. After solution the material in the flask shall be heated with full use of the heat until brown fumes are no longer apparent to the eye. Then 30 ml. of distilled water and a small amount (approximately 1 g.) of pure powdered talcum shall be added and the mixture boiled

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

vigorously for at least 10 min. The escaping vapors, when not too strongly acid, may be tested for nitrous acid with moist starch iodide paper. Boiling under these conditions should quickly give a negative test and need not be continued to dryness. After cooling, enough distilled water shall be added to bring the volume up to approximately 100 ml. The talc shall then be filtered off on an asbestos-padded Gooch crucible and washed thoroughly with hot distilled water. The entire filtrate with washings shall be transferred to a 1-liter volumetric flask, and, after cooling to 25 C., shall be made up to the mark with distilled water. The strength of this solution may be checked by electrolytic determination of the copper in tartrate-cyanide solution on a platinum dish cathode.[1]

(*b*) *Alternative Standard Copper Solution.*—For routine analysis the standard copper solution may be prepared by a more simple method as follows: 3.94 g. of clean, clear crystals of c.p. copper sulfate ($CuSO_4 \cdot 5\ H_2O$) shall be accurately weighed, then dissolved in about 100 ml. of distilled water containing 1 ml. of dilute sulfuric acid and diluted to 1 liter with distilled water in a volumetric flask. The strength of this solution may be checked by electrolysis as described in Paragraph (*a*) or by titration with a standard solution of sodium thiosulfate.

(*c*) *Diluted Standard Copper Solution.*—In a volumetric flask, 10 ml. of the standard copper solution, prepared as described in Paragraph (*a*) or (*b*), shall be diluted to 1 liter with copper-free distilled water at 25 C. One milliliter of this solution is equivalent to 0.00001 g. (10 micrograms) of copper.

(*d*) *Thiosulfate Solution.*—A solution of sodium thiosulfate of approximately 0.001 *N* strength shall be used. A standard solution of approximately 0.1 *N* strength shall first be prepared by dissolving about 25 g. of the crystalline sodium thiosulfate ($Na_2S_2O_3 \cdot 5\ H_2O$) and 2 g. of NaOH in 1 liter of distilled water. In a volumetric flask, 10 ml. of the 0.1 *N* sodium thiosulfate solution shall be diluted to 1 liter with distilled water. The 0.001 *N* sodium thiosulfate solution shall be standardized by taking an amount of the standard copper solution, approximately equivalent to the amount of copper expected in the sample, and treating it as outlined in the determination of copper, starting with the addition of 10 ml. of HCl (sp. gr. 1.19), Section 7 (*b*). The 0.001 *N* sodium thiosulfate solution is not very stable and shall be freshly prepared each day from the 0.1 *N* solution.

(*e*) *Starch Indicator Solution.*—This solution shall contain approximately 10 g. of soluble starch per liter of water. The water shall be boiled and the starch dissolved by adding it slowly, with rapid stirring, to the boiling solution. The starch solution shall be freshly prepared every two or three days.

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[1] A. W. Peters, "A Simple and Rapid Method of Electrolysis of Copper Solutions with Tartrate Cyanide Electrolyte," *Journal*, Am. Chemical Soc., Vol. 34, p. 426 (1912).

(*f*) *Bromine Water.*—The bromine water shall be made by adding an excess of bromine to distilled water, without adding a soluble bromide to increase its concentration. The presence of a soluble bromide leads to the formation of compounds of the nature of hypobromites which will liberate iodine and cause the results to be high.

Procedure

7. (*a*) *Ashing.*—The accurately weighed sample shall be cut into small pieces and placed in a No. ooA porcelain evaporating dish of 70-ml. capacity. The dish shall be placed on a Chromel triangle over a low flame from a bunsen burner protected from strong drafts. The sample shall then be ignited gently, care being taken that the material does not burn with a flame. The heating shall be continued until all the fiber has burned off leaving a carbonaceous skeleton. At this point the dish shall be allowed to cool and the residue shall be moistened carefully with five drops of HNO_3 (sp. gr. 1.42) and one drop of H_2SO_4 (sp. gr. 1.84). The heat shall again be applied gradually and raised until all carbon and tarry residue is burned off. The time consumed in ashing is usually about 20 min.

(*b*) *Titration.*—The ash shall be dissolved in 10 ml. of HCl (sp. gr. 1.19) by heating on a hot plate for 10 min. and then transferred to a beaker with 50 ml. of hot distilled water. Approximately 2 to 5 ml. of bromine water shall be added and the solution shall be boiled briskly until the excess bromine has been boiled off. In order to insure the complete removal of the bromine it is necessary to boil the solution down to about one third of its original volume. The solution shall be cooled slightly and NH_4OH (sp. gr. 0.90) added in slight excess. The solution shall then be boiled vigorously for a few minutes in order to coagulate the iron precipitate, expel the excess of ammonia, and destroy compounds which may be formed if all the bromine has not been expelled. The iron precipitate shall be filtered off through a thin asbestos mat and washed three or four times with a little hot distilled water. Dilute acetic acid shall then be added to the filtrate until the blue color just disappears or until the solution is just faintly acid to litmus paper. The solution shall then be heated to boiling for a few minutes more, cooled to tap water temperature, and 5 g. of potassium iodide (free from iodate) or an equivalent amount of a freshly prepared solution of potassium iodide shall be added. One milliliter of starch indicator shall be added and the solution shall be titrated immediately with 0.001 *N* sodium thiosulfate. It is necessary to stir the solution vigorously when titrating with the 0.001 *N* solution because diffusion is slower with such a dilute solution. It is advisable to titrate as quickly as possible to the end point. In some samples, if allowed to stand exposed to the air, a blue color will reappear after the titration is completed. This may be disregarded since an end point, permanent for 1 min., indicates that all of the copper has been titrated.

TENTATIVE DEFINITIONS AND TERMS RELATING TO TEXTILE MATERIALS[1]

A.S.T.M. Designation: D 123 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED AS TENTATIVE, 1921; ADOPTED IN AMENDED FORM, 1927; REVISED, 1930; REISSUED AS TENTATIVE, 1933; REVISED, 1934, 1935, 1936, 1937.

Abacá.—See Fibers, Vegetable.

African Sisal.—Under Fibers, Vegetable, see Sisal.

Airplane Fabric (Cotton).—A plain-weave fabric well woven, practically without imperfections, of mercerized two-ply yarns, width 36 to 56 in., maximum weight 4.5 oz. per sq. yd., and threads per inch warp and filling 80 to 84.

Ambari.—Under Fibers, Vegetable, see Kenáf.

Asbestos Roving.—Asbestos roving is composed of a mixture of chrysotile asbestos and cotton or other organic fiber.

Reinforced Asbestos Roving.—Reinforced asbestos roving is composed of a core of yarn consisting of organic fiber, covered with a mixture of chrysotile asbestos and organic fiber.

Asbestos Yarn.—Yarn consisting of: (1) asbestos fiber; (2) asbestos and organic fibers; (3) asbestos and organic fibers and wire; or (4) asbestos and organic fibers with an insert of cotton or other yarn reinforcement.

Plain Asbestos Yarn.—Yarn consisting of: (1) asbestos fiber, or (2) asbestos and organic fibers.

Metallic Asbestos Yarn.—Yarn consisting of plain asbestos yarn twisted with brass, copper or other fine wire. It is also termed "wire-inserted yarn."

Backing Yarn (Pile Fabrics).—Yarns that do not form the pile.

Bad Casts (Raw Silk).—Abruptly thickened places on the threads due to the cocoon filament not being properly attached to the thread, or made by adding more than one cocoon filament at a time.

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[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

These tentative definitions comprise a revision and combination of the former Standard Definitions of Terms Relating to Textile Materials (A.S.T.M. Designation: D 123 – 30) and Tentative Definitions of Terms Relating to Textile Materials (A.S.T.M. Designation: D 123 – 30 T), which definitions were accordingly discontinued in 1933.

Bagging.—A very heavy loosely woven fabric used for bale tares, etc., woven from heavy tight-twisted roving, consisting largely of reworked and waste fibers. Also known as "gunny."

Bahama Sisal.—Under Fibers, Vegetable, see Sisal.

Benares Sunn.—Under Fibers, Vegetable, see Sunn.

Bimlipatam Jute.—Under Fibers, Vegetable, see Kenâf.

Binder Twine.—A somewhat stiff, single strand yarn, 3 to 4 mm. in diameter, made of hard fibers (henequen, sisal, abacá, or phormium), used chiefly for binding bundles of grain, etc.

Boiled-Off Silk.—Silk with the gum (sericin) removed.

Bombay Aloe.—Under Fibers, Vegetable, see Cantara and Piteira.

Book-Fold.—The fabric is doubled selvage to selvage, then folded back and forth upon itself in predetermined lengths. When the piece is completed, the fold-edges on each side are folded to the center and the piece is folded once more upon itself so that the fold-edges are inside, forming a compact package as long as one-half the width of the goods.

Bristle.—A very coarse, stiff hair.

Broken Pick.—A streak across the width of the fabric caused by a completely or partially missing pick.

Brown Hemp.—Under Fibers, Vegetable, see Sunn.

Burlap (Jute).—A coarse, heavy, plain-weave fabric of coarse single yarn.

Cable Twist.—A twine, cord, or rope construction in which each successive twist is in the opposite direction to the preceding twist.

Camel's Hair.—The hair of the camel, particularly the soft downy fiber from the haunches and under parts of the camel.

Cantala.—See Fibers, Vegetable

Cellulose-Acetate Rayon.—See Fibers, Artificial.

China Grass.—See Fibers, Vegetable.

China Hemp.—Under Fibers, Vegetable, see Hemp.

Chingma.—See Fibers, Vegetable.

Cockled or Cockling.—A curliness or crimpiness appearing in fabrics when different sizes of yarn or varying tensions on the yarns are accidentally introduced.

Coir.—See Fibers, Vegetable.

Combing Wool.—See Wool, Combing.

Cordage Fibers.—See Fibers, Cordage.

Corkscrews (Raw Silk).—A place in the thread in which one or more cocoon filaments are longer than the remainder and give the appearance of a spiral form.

Heavy Corkscrews (Raw Silk).—Corkscrews which are more than twice the diameter of the thread.

Corkscrew Twist.—A place in the yarn or cord where an uneven twist gives a corkscrew-like appearance.

Cotton.—See Fibers, Vegetable.

Crimp.—The difference in distance between two points on a yarn as it lies in a fabric and the same two points when the yarn has been removed from the fabric and straightened, expressed as a percentage of the distance between the two points as the yarn lies in the fabric.

Cuprammonium Rayon.—See Fibers, Artificial.

Cut (Asbestos).—A term indicating the number of an asbestos yarn. The word "cut" preceded by a number indicates, in multiples of 100 yd., the yardage per pound of single-ply yarn. For example, "5 cut" indicates that a pound of single-ply yarn so designated measures approximately 500 yd.; "10 cut" indicates a yarn that measures approximately 1000 yd. per lb.

(Woolen).—See Yarn Number (Woolen—Cut).

Da or Dha.—Under Fibers, Vegetable, see Kenâf.

Daisee Jute.—Under Fibers, Vegetable, see Jute.

Defective Splice.—A splice having irregular final twist, untrimmed ends, or excessive thickness or thinness.

Denier.—A unit of weight (0.05 g.) used to express the number of silk and rayon yarns. The weight in units of 0.05 g. of a 450-m. skein at standard conditions is the denier of silk and rayon (exclusive of spun silk and staple rayon) yarn. It is also equal numerically to the weight in grams of a 9000-m. skein at standard conditions.

Dungaree (Cotton).—A heavy cloth woven in the 2/1 twill weave from dyed warp and filling yarns, in narrow widths, usually 28 in.; approximate number of threads per inch, warp 68 and filling 52; yarn number both warp and filling 20's.

End.—An individual warp yarn.

Fell Jackets.—See Jackets.

Fiber.—A slender, elongated filament, such as rayon, silk, or wool; or a single elongated cell, like cotton or kapok; or a slender strand composed of aggregations of elongated cells like flax, jute and sisal.

Fibers, Artificial:

Cellulose-Acetate Rayon.—Filaments composed of an acetic ester of cellulose which has been coagulated or solidified from its solution.

Cuprammonium Rayon.—Filaments composed of a regenerated cellulose which has been coagulated or solidified from a solution of cellulose in ammoniacal copper oxide.

Nitrocellulose Rayon (Chardonnet).—Filaments composed of a regenerated or denitrated cellulose which has been coagulated or solidified from a solution of nitrated cellulose.

Viscose Rayon.—Filaments composed of a regenerated cellulose which has been coagulated or solidified from a solution of cellulose xanthate.

Fibers, Asbestos:

Amosite.—A mineral fiber occurring as a silicate of iron produced principally in Africa. Amosite is a coarse brown or gray fiber. It has little application in the textile industry, except as a filler for asbestos insulating products.

Chrysotile.—A mineral fiber occurring as a silicate of magnesia in connection with serpentine rock, produced extensively in Canada, Africa, and Russia and to smaller extent in the United States, Italy, Australia and China. Chrysotile is found as a compact fibrous stone in various shades of green and yellow which, when crushed, separates into fine white fibers. The division into fibers can be carried to extreme fineness and the ultimate fiber is regarded as a chain of molecules.

Crocidolite.—A mineral fiber occurring as a silicate of iron produced principally in Africa. Crocidolite found as a stone is blue in shade and is reduced to fibrous form by mechanical process of separation.

Fibers, Cordage.—Coarse hard fibers, such as abacá, sisal, henequen, cantala, phormium, palma and piteira (see Fibers, Vegetable), used in the manufacture of ropes and coarse twines. Generally too coarse and stiff to be woven into fabrics.

Fibers, Hard or Leaf.—Rather stiff elongated strands from leaves like sisal, henequen, and piteira (see Fibers, Vegetable), or leaf stems like abacá, used chiefly in coarse twines, ropes and heavy cordage.

Fibers, Soft or Bast.—Flexible elongated strands from the inner bark of plants like flax, hemp, jute and ramie, used in twines, nets and woven fabrics.

Fibers, Textile.—Slender filaments or strands that cling together, enabling them to be spun into yarns of appreciable strength and sufficiently pliable to be woven or knitted into fabrics. Essential requisites are tensile strength, length of at least 5 mm. and more than 100 times the diameter, pliability and cohesiveness;

Fibers, Textile (Continued):

desirable, but not essential, properties are elasticity, fineness, uniformity, durability and luster. Textile fibers include cotton, flax, jute, and many other fibers of vegetable origin; silk, wool and various hairs of animal origin; rayon and allied synthetic or artificial fibers; and asbestos of mineral origin.

Fibers, Vegetable:

Abacá (a-ba-ká).—A hard fiber, rather coarse, firm texture, cream-white to brown in color, 150 to 350 cm. long, strong and durable from the leaf stems forming the trunk of the abacá plant, *Musa textilis,* cultivated in the Philippine Islands, Sumatra, Java and Borneo; used in ropes and coarse twines and cordage, especially marine cordage, oil-well cables, and binder twine. Called "Manila hemp" in the market, but quite unlike true hemp, also called "Cebu hemp" and "Davao hemp."

Cantala.—A hard fiber, finer, softer and weaker than sisal, yellow-white to brownish white, 75 to 150 cm. long, from leaves of the cantala plant, *Agave cantala,* cultivated in the Philippine Islands, Java, Sumatra and India; used in coarse twines including binder twine. Called "Manila maguey" and "Cebu maguey" in market quotations for water-retted, hand-cleaned cantala of the Philippine Islands.

China Grass.—Ramie fiber separated from the stalk and the thin outer bark, but not degummed; the form in which it is usually exported from China.

Chingma (Ching′-mah).—A rather coarse soft fiber, usually flaggy and fuzzy, straw-white, from the inner bark of the chingma plant, *Abutilon theophrasti,* cultivated in northern China and Japan. Called "China jute" and "Tientsin jute," but it is not a true jute.

Coir.—A hard fiber, usually brown, 10 to 25 cm. long, from the outer husk of the cocoanut, *Cocos nucifera,* produced chiefly in Ceylon and southern India; used in door mats and as a stuffing fiber in upholstery.

Cotton.—Fine, one-celled, twisted fibers, white or in some types tawny, brown or green, 20 to 50 mm. long, growing on the seeds of several different species of the genus *Gossypium*; used more than all other vegetable fibers, and for a wider range of purposes including yarns, twines, woven and knit goods, upholstery stuffing and packing.

Fibers, Vegetable (Continued):

American short-staple upland cotton.—Fiber 20 to 27 mm. long, from *Gossypium hirsutum*, cultivated in the southern part of the United States and introduced into nearly all cotton-growing countries.

American long-staple upland cotton.—28 to 40 mm. long, from selected varieties of *Gossypium hirsutum*, cultivated largely in the Yazoo-Mississippi delta. Also called "delta cotton."

Arizona Egyptian cotton.—Fiber 35 to 45 mm. long, from modified forms of *Gossypium barbadense*, cultivated in Arizona and southern California, includes "Pima cotton."

Egyptian cotton.—Fiber 30 to 40 mm. long, white or in some varieties light brown, from modified forms of *Gossypium barbadense*, cultivated chiefly in lower Egypt, includes "Sakellarides," "Zagora," etc.

India cotton.—Short staple, 18 to 25 mm. long, from *Gossypium neglectum*, cultivated in India.

Levant cotton.—Short staple, 18 to 25 mm. long, from *Gossypium herbaceum*, cultivated in Asia Minor and countries bordering the Mediterranean Sea.

Rough Peruvian cotton.—A rather harsh, wiry cotton, from *Gossypium peruvianum*, cultivated in Peru, used chiefly for mixing with wool.

Sea island cotton.—Fine fiber, 30 to 50 mm. long, from *Gossypium barbadense*, cultivated chiefly in the West Indies, used in the finest cotton yarns.

Flax.—A soft, fine fiber, dark gray, bluish green, brown or cream white, 40 to 100 cm. long, from the inner bark of the fiber flax plant, *Linum usitatissimum*, cultivated chiefly in northern Europe; used in linen thread, shoe thread, seine twine, sacking twine, laces, table linen, handkerchiefs, towels and in all true linens.

Courtrai flax.—Flax fiber of superior quality, very carefully prepared from straw retted in the River Lys, or most of it in recent years tank-retted in the vicinity of Courtrai, Belgium.

Fibers, Vegetable (Continued):

Dew-retted flax (Slanetz of Russia).—Flax fiber prepared by spreading the plants on the ground and exposing to the weather for retting.

Flax tow.—Short or tangled fiber beaten out in scutching—often called scutching tow; or combed out in hackling and called machine tow; used in flax yarns and twines. (This flax tow is distinctly different from upholstery flax tow.)

Flemish flax.—Flax produced in Flanders (northeastern France and Belgium).

Hackled flax (also called "dressed flax" or "dressed line").—Flax fiber, which after being scutched, has been cleaned further by combing over hackles.

Irish flax.—Flax fiber produced in northern Ireland, mostly retted in bogs, yielding a very strong fiber.

Risten (*R* in market quotations).—The standard grade of flax on which other grades of the Baltic countries are based.

Water-retted flax (Motchenetz of Russia).—Flax fiber, light cream-white to gray in color, prepared by retting the plants in water.

Hemp.—A soft fiber, dark steel gray to cream-white, usually in narrow flat ribbons, 100 to 250 cm. long, from the inner bark of the hemp plant, *Cannabis sativa*, cultivated in Italy, Soviet Republic, Yugoslavia, Hungary, China, Japan, Chile, Wisconsin, Illinois and Kentucky; used in America in commercial twines, marlines, halyards and tarred rigging.

Hemp line.—Hemp fiber, scutched and prepared so that the fibers lie straight and parallel.

Hemp tow.—Short or tangled hemp fiber, beaten out in scutching the long fiber, or hemp fiber produced from short or tangled stalks.

Italian hemp.—Hemp fiber produced in Italy, mostly water-retted and well prepared and graded.

Kentucky rough prime.—The best quality of Kentucky dew-retted hemp, broken on hand brakes, but not hackled.

Fibers, Vegetable (Continued):

Kentucky single-dressed.—Kentucky hemp further prepared by drawing it over a coarse hackle.

Russian hemp.—Hemp fiber chiefly dew-retted and prepared by hand in the Soviet Republic.

Wisconsin hemp.—Hemp fiber produced in Wisconsin by dew-retting and breaking and scutching by machinery.

Henequen (hen-e-ken′).—A hard fiber, rather coarse, of firm texture, light yellow or sometimes reddish yellow, cylindrical, 75 to 150 cm. long, from the leaves of the henequen plant, *Agave fourcroydos,* cultivated in the Yucatan peninsula, and in Tamaulipas in Mexico and in Cuba; used chiefly in binder twine and in other coarse twines and small ropes.

Istle (eest′-ley).—Hard fibers, light yellow, 20 to 50 cm. long, cylindrical and stiff, from the leaves of two closely related species of *Agave,* growing without cultivation in Mexico, used chiefly in brushes, and to a less extent in twines; used in Mexico for handmade twines and coarse sacks. Also called "Tampico" and "Mexican Fiber."

Jute.—Soft fibers, light yellow to nearly white or bright copper to deep slate, changing in age to dingy brown or gray, 100 to 500 cm. long, averaging about 240 cm. exclusive of butts. from the inner bark of the round-pod jute, *Corchorus capsularis,* and the long-pod jute, *Corchorus olitorius,* cultivated in India, chiefly in Bengal, and to a very limited extent elsewhere in southern Asia and in tropical Africa; used in twines, gunnies, burlaps, hessians, carpet yarns, sacks for sugar, coffee, grain, feed, fertilizer and wool, and backing for linoleum and oilcloths.

Jute butts.—The flaggy lower ends of jute fiber, 10 to 25 cm. long, cut off in preparing jute for market, used in twines and coarse bagging, especially in covering for bales of cotton.

Daisee jute (also spelled *desi* and *deshi,* meaning "local").—Fiber of long-pod jute, *Corchorus olitorius,* grown in the brackish soils in the region of Calcutta, generally ranging from copper brown to slate gray.

Tossa jute.—Fiber of long-pod jute, *Corchorus olitorius,* grown on uplands in eastern India; pearl gray to deep slate color or copper brown.

Fibers, Vegetable (Continued):

Kapok.—Stuffing fiber, composed of single cells 20 to 30 mm. long, white gray or tawny in color, produced in the seed pods of the kapok tree, *Ceiba pentandra*, and closely allied species of the bombax family, resistant to the absorption of moisture, resilient and buoyant; used as stuffing in pillows, mattresses and life preservers and for temperature and sound insulation.

Kenáf.—A soft jute-like fiber, light yellow to gray, flaggy at base, 150 to 250 cm. long, from the inner bark of the kenáf plant, *Hibiscus cannabinus*, cultivated most extensively in Persia and Trans-Caspian Russia. Called "Da" in French West Africa, "Gombo" in Portuguese Africa, "Ambari" and "Deccan Hemp" in India, "Bimlipatam" in Madras Presidency; used in twines and for purposes similar to jute.

Mescal Maguey.—A hard fiber, light straw-yellow, slightly finer and softer than sisal, 80 to 150 cm. long, from leaves of the mescal maguey plant, *Agave pseudotequilana*, cultivated in western Mexico; used in twines and cordage.

Palma.—A hard fiber nearly white or light brownish yellow, often with some coarser dark brown curved ends, 25 to 50 cm. long, from the leaves of a tree yucca, *Samuela carnerosana*, growing on high mountains in northern Mexico; used for twines, especially "paper twines." Also called "palma istle," but quite unlike istle fibers.

Phormium (for′-mi-um).—A hard fiber, reddish yellow or nearly white, finer and softer than abacá, 150 to 300 cm. long, from the leaves of the harakeke lily, *Phormium tenax*, growing wild and cultivated in New Zealand, and cultivated in St. Helena; used chiefly in hard fiber twines, including binder twine. Called "New Zealand hemp" and "New Zealand flax" but the fiber is unlike hemp or flax.

Piteira (pe-ter′-a).—A hard fiber, nearly white, and finer and softer than other hard fibers as it is prepared in Mauritius, 150 to 250 cm. long, from the leaves of the giant cabuya, *Furcraea gigantea*, native in eastern Brazil and cultivated in Mauritius, India and Natal; used chiefly for mixing with other fibers to improve the color of ropes. Called "Mauritius hemp," but it is not a true hemp and is produced in other countries besides Mauritius.

Fibers, Vegetable (Continued):

Ramie.—A soft fiber, parchment-like, light yellow or greenish and stiff in the form called "China grass," white, soft and slightly coarser than flax when degummed and bleached, very strong and durable, from the inner bark of the ramie plant, *Boehmeria nivea*, cultivated in China and Japan; used in China for dress goods and "grass cloth," in Japan for seine twine and mosquito nets and in Germany for shoe thread, trimmings, tapestries, and various woven fabrics. Also called "rhea."

Sisal.—A hard fiber, of firm texture, light yellow or nearly white sometimes slightly green, very strong and durable, 100 to 180 cm. long, from the leaves of the sisal plant, *Agave sisalana*, native in Yucatan, cultivated in East Africa, Java, Sumatra, Haiti and the Bahamas; used in hard fiber twines including binder twine and also in coarse cordage.

Bahama Sisal.—Retted in sea water and cleaned by hand; is finer, softer, weaker and lacking in luster.

Sunn.—A soft fiber, gray or brown with a wide range of color and quality due to differences in preparation, 50 to 150 cm. long, from the inner bark of the sunn plant, *Crotalaria juncea*, cultivated in India; used in twines. Also called "Sunn hemp," "India hemp," "Bombay hemp," "Brown hemp," and various sorts by the names "Benares," "Itarsi," "Jubbulpore," "Madras" and "Sewnee."

Filament.—A fine fiber or fibril.

Flax.—See Fibers, Vegetable.

Float (Defect).—A place in the fabric where warp or filling threads extend unbound over threads with which they should be interlaced.

Gage.—A measure of the fineness of knit fabric as determined by the closeness of the wales, that is, the longitudinal rows of loops. It is expressed by the number of wales or needles in one and one half inches.

NOTE.—The word "gage" is also used as an expression of thickness.

Gombo Hemp.—Under Fibers, Vegetable, see Kenáf.

Grease Wool.—See Wool, Grease.

Fibers, Vegetable (Continued):

Grenadine Yarn (Silk).—A yarn composed of more than one strand, each individual strand of which has been doubled with a reverse twist, each twist having a minimum of approximately the following number of turns per inch depending on the number of strands used:

STRANDS	MINIMUM NUMBER OF TURNS
2 ($\frac{13}{15}$ denier or equivalent)	32
3	30
4	28

NOTE.—When more than 4 strands are used, the minimum number of turns is decreased in accordance with the above proportion.

Hairiness (Raw Silk).—The condition of the thread when there are numerous loose ends of cocoon filaments projecting from the thread.

Hawser Twist (Cotton).—A twine, cord or rope construction in which the first and second twists are in the same direction and the third twist in the opposite direction.

Heavy Corkscrews.—See Corkscrews.

Heavy Goods.—Name applied in Calcutta quotations to material called "sacking" in the United States.

Hemp.—See Fibers, Vegetable.

Henequen.—See Fibers, Vegetable.

Hessians.—Name applied in United Kingdom, India, and on the Continent to material called "burlap" in the United States.

Holland Finish (Cotton).—A glazed or unglazed finish, consisting of oil and a filling material, applied to cotton fabrics to make them opaque or semi-opaque; resembles a beetled linen fabric formerly known as "Holland."

Indian Hemp.—Under Fibers, Vegetable, see Sunn.

Istle.—See Fibers, Vegetable.

Italian Hemp.—Under Fibers, Vegetable, see Hemp.

Jackets (woven or felted).—A textile product, woven or pressed into tubular or sleeve form, ready for covering and shrinking on a machine roll.

Jubbulpore Hemp.—Under Fibers, Vegetable, see Sunn.

Jute.—See Fibers, Vegetable.

Jute Butts.—See Fibers, Vegetable.

Kapok.—See Fibers, Vegetable.

Kenaf.—See Fibers, Vegetable.

Kink (fabric).—A place in the fabric where a slack strand has twisted on itself, forming a loop in the yarn or cord.

Knot.—A joining by tying of threads together.

Long Knots (Raw Silk).—Knots which have loose ends from 3 to 25 mm. (⅛ to 1 in.) in length.

Very Long Knots (Raw Silk).—Knots which have loose ends exceeding 25 mm. (1 in.) in length.

Laid Fabric (Cotton).—A fabric made without filling yarn, the parallel warp yarns being held together by means of rubber latex or other binding material.

Lea (Cotton).—A 120-yd. skein of yarn containing 80 turns, that is, each 1½ yd. in length.

(Linen).—See Yarn Number (Linen).

Linen.—Under Fibers, Vegetable, see Flax.

Loop-Knot.—A snarl or curl produced by a filling yarn coiling upon itself.

Loops (Raw Silk).—Small open places in the thread due to the excessive length of one or more cocoon filaments.

Long Loops.—Loops which exceed 1 cm. (½ in.) in length.

Long Knot.—See Knot.

Maguey.—Under Fibers, Vegetable, see Cantala and Mescal Maguey.

Manila Hemp.—Under Fibers, Vegetable, see Abacá.

Mauritius Hemp.—Under Fibers, Vegetable, see Piteira.

Mescal Maguey.—See Fibers, Vegetable.

Metallic Asbestos Yarn.—See Asbestos Yarn.

Mexican Fiber.—Under Fibers, Vegetable, see Istle.

Mexican Sisal.—Under Fibers, Vegetable, see Henequen.

Mispick.—A pick not properly interlaced.

Mohair.—The hair of the Angora goat.

Moisture Content (Moisture, Percentage of Moisture).—The moisture present in a textile material expressed as a percentage of the original weight as determined by definite prescribed methods.

Moisture-Free Weight.—The weight of a textile material determined after drying to constant weight by definite prescribed methods.

Moisture Regain.—The moisture present in a textile material expressed as a percentage of the moisture-free weight, as determined by definite prescribed methods.

Natal Hemp.—Under Fibers, Vegetable, see Piteira.

Neatness (Raw Silk).—An expression of the degree of freedom of the raw silk thread from loops, nibs, and hairiness as defined.

New Zealand Flax.—Under Fibers, Vegetable, see Phormium.

Nibs (Raw Silk).—Small thickened places less than 3 mm. ($\frac{1}{8}$ in.) in length.

Nitrocellulose Rayon.—See Fibers, Artificial.

Number of Yarn.—See Yarn Number.

Off-Square.—The difference between the percentage of warp crimp and the percentage of filling crimp.

Organzine Yarn.—Two or more raw silk strands twisted in the singles doubled and twisted in the reverse direction in the ply. Turns per inch in the singles and ply usually within the limits of 10 to 20 turns; generally used as warp.

Palma.—See Fibers, Vegetable.

Philippine Maguey.—Under Fibers, Vegetable, see Cantala.

Phormium.—See Fibers, Vegetable.

Pick.—An individual filling yarn.

Pile.—The raised portion of the fabric which masks the other yarns and forms the wearing surface of the pile fabrics and pile floor coverings.

Pile Fabric.—A fabric the surface of which is formed by loops or tufts (cut loops) of the pile yarn.

Pile Yarn.—The yarn of which the pile is formed.

Pitch (Pile Floor Coverings).—The number of pile ends per inch of width.

Piteira.—See Fibers, Vegetable.

Plain Weave.—A fabric pattern in which each yarn of the filling passes alternately over and under a yarn of warp and each yarn of the warp passes alternately over and under a yarn of the filling.

Pulled Wool.—See Wool, Pulled.

Ramie.—See Fibers, Vegetable.

Raw Silk.—Silk as it has been reeled from the cocoon.

Rayon.—A generic term for filaments made from various solutions of modified cellulose by pressing or drawing the cellulose solution through an orifice, and solidifying it in the form of a filament.

NOTE.—Rayon as commercially manufactured is produced by four different processes. The resulting rayons from each process while similar in appearance differ amongst themselves and with each other to a more or less degree according to the differences in their physical and chemical properties. The different type rayons are known as follows:

(*a*) Nitrocellulose Rayon (Chardonnet)
(*b*) Viscose Rayon
(*c*) Cuprammonium Rayon
(*d*) Cellulose-Acetate Rayon

Reed Marks.—Marks or lines warpwise of the cloth, usually caused by too many or too few warp yarns being drawn into a dent, a faulty setting of the loom, or imperfections in the reed.

Regain.—See Moisture Regain.

Reinforced Asbestos Roving.—See Asbestos Roving.

Rows (Pile Floor Coverings).—The number of rows of tufts per inch in the direction of the warp.

Run (Woolen).—See Yarn Number (Woolen Run).

Russian Hemp.—Under Fibers, Vegetable, see Hemp.

Sewed Yarn.—A yarn in the fabric interwoven by hand.

Shoe-Fold.—The fabric is folded from both ends into twelve or sixteen folds to the piece, the length of the fold depending upon the length of the piece.

Shot (Pile Floor Coverings).—The number of filling yarns per row of tufts.

Shrinkage.—The loss of weight or dimension in any given process.

Sisal.—See Fibers, Vegetable.

Size.—See Yarn Number.

Slub.—See Slug (General).

Slug (Raw Silk).—A thickened place several times the diameter of the thread, 3 mm. (⅛ in.) or over in length.

Long or Large Slug (Raw Silk).—A slug which exceeds 1 cm. (½ in.) in length or which is very much larger in diameter than the thread.

(General).—An abruptly thickened place in the yarn or a bunch of lint entangled in the yarn, cord, or fabric.

Smash.—A place in the fabric where a number of warp yarns or cords have been broken, usually due to the shuttle getting caught in the shed during weaving, or to faulty harness motion.

Spun Rayon.—Yarn made from cut rayon filaments, the cut filaments being twisted and drawn out into a yarn by usual spinning processes.

Spun Silk.—Silk yarn consisting of short filaments obtained from silk wastes spun in a similar manner to worsted yarn.

Standard Atmosphere (Textiles).—Air maintained at a relative humidity of 65 per cent at 70 F.

Standard Moisture Regain.—An arbitrary figure formally adopted for the regain of any specific textile material to be used in calculating the standard weights of shipments or deliveries of textiles.

Standard Weight.—The moisture-free weight of a textile material plus the weight corresponding to its standard moisture regain.

Suint (Wool).—Excretions from the sweat glands of the sheep, principally potash salts.

Sunn.—See Fibers, Vegetable.

Tampico Fiber.—Under Fibers, Vegetable, see Istle.

Thick and Thin Places (Major Defects).—Places in the fabric where for 1 in. or more the count varies more than a specified percentage from the specified count.

(Minor Defects).—Places in the fabric where for less than 1 in. the count varies more than a specified percentage from the specified count.

Thread (Silk).—The ply of yarns is sometimes referred to as the number of thread, as 2 thread for a 2 ply.

Thrown Silk.—Raw silk that has been twisted, or doubled and twisted.

Tientsin Jute.—Under Fibers, Vegetable, see Chingma.

Tire Builder Fabric.—A fabric consisting of hawser cord yarn in the warp with single yarn filling at intervals to keep the warp threads together.

Tossa Jute.—Under Fibers, Vegetable, see Jute.

Tram Yarn (Silk).—Raw silk threads doubled and twisted with a low number of turns per inch, generally used as filling.

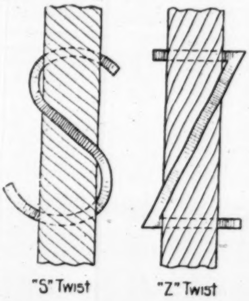
Tula Istle.—Under Fibers, Vegetable, see Istle.

Twill Weave.—A weave which produces pronounced lines running diagonally across the fabric.

Warp Face Twill.—A twill weave in which the warp threads produce the diagonal effect.

Twine (Not Binder Twine).—A cord made up of two or more yarns of medium twist with ply twist in the opposite direction.

Twist, Direction of.—A yarn or cord has **S** twist if, when held in a vertical position, the spirals conform in slope to the central portion of the letter "S," and **Z** twist if the spirals conform in slope to the central portion of the letter "Z."



Typp.—A method of yarn numbering. The number of thousands of yards per pound.

Viscose Rayon.—See Fibers, Artificial.

Waste:

(Card).—Short fluffy waste thrown out of the carding machine.

(Hard).—Waste from reeling, warping, winding, weaving, and cop bottoms.

(Lap).—Waste of long fibers, formed in drawing and spinning processes by the breaking of the ends between the front rollers and the bobbins. This may be re-processed.

(Soft).—Waste from slubbing, roving, and wool tops.

(Spinners).—Broken and tangled threads and lengths of yarn left on bobbins and spinning machinery in the manufacture of yarn.

(Thread).—The waste left on bobbins or collected during spinning and weaving.

(Thrum).—The threads of woven out warps cut from warp beams. This waste is often tightly twisted and must be reworked before it can be used again.

(Sweeping).—The sweeping from the floors of the various rooms in the mill. This is usually of a short and fluffy character.

(Bur-Wool).—The waste removed by the bur guards of cards or bur pickers having a very short fiber and full of burs or seeds. It naturally varies according to the wool from which the burs are taken.

Webbing, Elastic.—A strong narrow fabric made with rubber threads as part of warp or filling or of both.

Wool, Combing.—Wool that is strong and strictly of combing length, the lengths differing for the different grades as follows: Fine 50 mm. (2 in.) or more; half blood 56 mm. (2¼ in.) or more; three-eighths blood 62 mm. (2½ in.) or more; quarter blood 68 mm. 2¾ in.) or more; low quarter blood 75 mm. (3 in.) or more. Wools of the grades common and braid, when normally grown for a period of one year, are always of combing lengths.

Wool, Grease.—Wool as it comes from the living sheep, not washed or scoured.

Wool, Pulled.—Wool taken from the skin of slaughtered sheep's pelt by slipping, sweating or the use of depilatory.

Wool (Yarn Number).—See Yarn Number (Woolen and Worsted).

Woolen.—Yarn spun from wool fibers which have been carded but not combed or gilled. Cloth woven from this yarn.

Wrong Draw.—A defect in a fabric due to the warp and filling not interlacing in accordance with the prescribed weave or design and caused when one or more warp yarns have been drawn through the wrong harness.

Yarn.—An assemblage of fibers or filaments of animal, mineral, or vegetable origin, alone or in combination, either natural or manufactured, twisted or laid together to form a strand or group of strands for use in weaving, knitting, or forming in any manner into textile materials.

Yarn Number (Asbestos).—See Cut (Asbestos).

(Cotton).—The number of 840-yd. hanks (cotton system) or the number of thousands of yards (Typp System) contained in 1-lb. avoirdupois (7000 grains) of the yarn at standard conditions. The number of folded or plied yarns is that of the single yarn from which they are spun.

(Jute).—The weight in pounds of a spindle of 14,400 yd. of yarn at standard conditions expressed as "pounds per spindle."

(Linen).—The number of 300-yd. hanks contained in 1 lb. avoirdupois (7000 grains) of the yarn at standard conditions. The number of folded or plied yarns is that of the single yarn from which they are spun. Also known as "lea."

(Raw Silk).—See Denier.

(Rayon).—See Denier.

(Spun Rayon).—The number of 840-yd. hanks contained in 1 lb. avoirdupois (7000 grains) of the yarn at standard conditions. The number of folded or plied yarns is that of the single yarn from which they are spun.

(Spun Silk).—The number of 840-yd. hanks contained in 1 lb. avoirdupois (7000 grains) of the yarn at standard conditions. The number of folded or plied yarns is that of the finished yarn.

(Woolen—Cut).—The number of 300-yd. "cuts" or hanks in 1 lb. avoirdupois (7000 grains) of the yarn at standard conditions.

(Woolen—Run).—The number of 1600-yd. hanks in 1 lb. avoirdupois (7000 grains) of the yarn at standard conditions.

(Woolen and Worsted—Typp System).—The number of a woolen or worsted yarn shall be the number of thousands of yards per pound.

(Worsted).—The number of 560-yd. hanks in 1 lb. avoirdupois (7000 grains) of the yarn at standard conditions. The number of folded or plied yarns is that of the single yarn from which they are spun.

Yarn Size.—See Yarn Number.

TENTATIVE SPECIFICATIONS

FOR

CAUSTIC SODA[1]

A.S.T.M. Designation: D 456 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover material suitable for various washing, cleaning and scouring processes, with or without soap as conditions demand, and where a strongly alkaline material is desired.

Caustic Soda

2. Caustic soda shall be furnished in air-tight containers in flake, coarsely powdered or solid form as specified by the purchaser.

Chemical Composition

3. Caustic soda, as received, shall conform to the following requirements as to chemical composition:[2]

Total alkalinity as Na_2O, min., per cent	75.5
Sodium hydroxide (NaOH), min., per cent	96
Carbonate as Na_2CO_3, max., per cent	2

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-12 on Soaps and Detergents.

[2] Until standard methods are available, reference may be made to the Proposed Methods of Chemical Analysis of Special Detergents which are under consideration by Committee D-12 on Soaps and Detergents and have been printed as information in Appendix II to the 1937 report of the committee, see p. 477.

TENTATIVE SPECIFICATIONS
FOR
MODIFIED SODA (SESQUICARBONATE TYPE)[1]

A.S.T.M. Designation: D 457 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover material suitable for various washing, cleaning and scouring processes, with or without soap as conditions demand, and where a mildly alkaline material is desired.

Modified Soda

2. Modified soda (sesquicarbonate type) shall be a white, uniform powder or crystal, as specified by the purchaser, composed of sodium carbonate and sodium bicarbonate.

NOTE.—This product is commonly known as laundry soda.

Chemical Composition

3. Modified soda (sesquicarbonate type), as received, shall conform to the following requirements as to chemical composition:[2]

	MINIMUM	MAXIMUM
Total alkalinity as Na_2O, per cent	39	43
Sodium bicarbonate ($NaHCO_3$), per cent	35	50
Sodium carbonate (Na_2CO_3), per cent	35	50
Matter insoluble in water, per cent	..	0.1

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-12 on Soaps and Detergents.

[2] Until standard methods are available, reference may be made to the Proposed Methods of Chemical Analysis of Special Detergents which are under consideration by Committee D-12 on Soaps and Detergents and have been printed as information in Appendix II to the 1937 report of the committee, see p. 477.

TENTATIVE SPECIFICATIONS
FOR
SODA ASH[1]

A.S.T.M. Designation: D 458 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover material suitable for various washing, cleaning and scouring processes, with or without soap as conditions demand, and where a moderately strong alkaline material is desired.

Soda Ash

2. Soda ash shall be a high-grade anhydrous sodium carbonate in powdered form and shall be of the type specified by the purchaser.

NOTE.—These specifications cover the 58 per cent grade of soda ash (Na_2O).

Chemical Composition

3. Soda ash, after drying for 1 hr. at 155 C., shall conform to the following requirements as to chemical composition:[2]

Total alkalinity as Na_2CO_3, min., per cent	99.16
Sodium hydroxide ($NaOH$), max., per cent	0.1
Sodium bicarbonate ($NaHCO_3$), max., per cent	0.5
Matter insoluble in water, max., per cent	0.25

Loss on Heating

4. (*a*) At the place of manufacture, the loss in weight on heating 2 g. of the material at 150 to 155 C. for 1 hr. shall not exceed 1 per cent.

(*b*) When the sample is taken at other than the place of manufacture, it shall be recognized that the soda ash is hygroscopic. Hence from the percentage loss in weight on heating, there shall be deducted the percentage gain in net weight of the package as sampled over the net shipping weight, to arrive at the percentage loss in weight on heating at the place of manufacture.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-12 on Soaps and Detergents.

[2] Until standard methods are available, reference may be made to the Proposed Methods of Chemical Analysis of Special Detergents which are under consideration by Committee D-12 on Soaps and Detergents and have been printed as information in Appendix II to the 1937 report of the committee, see p. 477.

Types

5. Types of soda ash shall be designated as follows on the basis of the density as received:

	Apparent Volume of 30 g., ml.		Apparent Density, lb. per cu. ft.	
	Min.	Max.	Min.	Max.
Light (Type A)	50	65	29	37
Extra-light (Type B)	75	95	20	25

TENTATIVE SPECIFICATIONS
FOR
MILLED TOILET SOAP[1]

A.S.T.M. Designation: D 455 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Scope

1. These specifications cover milled toilet soap.

General Requirements

2. Milled toilet soap shall be a high-grade, milled cake soap, either colored or uncolored, mildly perfumed unless otherwise specified, as free as possible from water, thoroughly saponified, and well compressed in firm, smooth cakes of a size and shape specified in the contract. It should lather freely when used with cold, soft water.

Chemical Composition

3. Milled toilet soap shall conform to the following requirements as to chemical composition. The percentage of matter volatile at 105 C. shall be calculated on the basis of the soap as received, but all other constituents shall be calculated on the basis of material containing 15 per cent of matter volatile at 105 C.

Moisture and matter volatile at 105 C., max., per cent	15[a]
Sum of free alkali, total matter insoluble in alcohol and sodium chloride, max., per cent	1.7
Free alkali, calculated as NaOH, max., per cent	0.1
Matter insoluble in water, max., per cent	0.4
Unsaponified saponifiable matter (free fat), max., per cent	0.3
Rosin, sugar and foreign matter	none
Anhydrous soap, min., per cent	83.0

[a] Deliveries which yield more than 15 per cent volatile matter will be rejected without further test.

Basis of Purchase

4. Milled toilet soap is subject to a possible gain or loss of weight, depending on atmospheric or storage conditions, or both, or on packaging, as a result of fluctuation in the moisture content. Changes in the moisture

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-12 on Soaps and Detergents.

content result in a corresponding change in the percentage of total solids or anhydrous soap, or both.

(*a*) The material shall be purchased by net weight provided the matter volatile at 105 C. is neither above nor below 15 per cent.

(*b*) Deliveries containing more than 15 per cent of matter volatile at 105 C. shall be rejected without further test.

(*c*) On deliveries containing less than 15 per cent of matter volatile at 105 C. settlement shall be made on the basis of a product containing 15 per cent moisture, that is, 0.85 lb. of non-volatile matter shall be considered 1 lb. of soap.

When the material conforms to the specifications on a calculated 15 per cent moisture and volatile matter basis the net weight of the material to be paid for shall be calculated as follows:

$$\begin{array}{l}\text{Net weight of material to} \\ \quad\text{be paid for on 15 per} \\ \quad\text{cent moisture and vol-} \\ \quad\text{atile matter basis}\end{array} = \frac{\text{Net weight as received} \times (100 - \text{percentage of loss at 105 C.})}{85}$$

In the event that the above calculation results in a figure greater than 100 per cent due to low moisture content at the time of cutting, weighing and packing, the seller waives the right to invoice the purchaser for any amount in excess of 100 per cent.

Method of Sampling and Testing

5. The material shall be sampled and analysed in accordance with the Tentative Methods of Sampling and Chemical Analysis of Soaps and Soap Products (A.S.T.M. Designation: D 460 – 37 T) of the American Society for Testing Materials.[1] The purchaser reserves the right to use any additional available information to ascertain whether the material conforms to the specifications.

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[1] See p. 1266.

TENTATIVE METHODS OF SAMPLING AND CHEMICAL ANALYSIS OF SOAPS AND SOAP PRODUCTS[1]

A.S.T.M. Designation: D 460 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

These methods are identical in substance with the standard methods of the American Oil Chemists Society which were developed by the Committee on Soap Analysis A-1 of that Society, and with those of the American Chemical Society.

Scope

1. (*a*) These methods describe procedures for the sampling and chemical analysis of cake, powdered, flake, liquid, and paste soaps, and soap products.

(*b*) The procedures for sampling and the analytical methods for soaps and soap products appear in the following order:

		SECTION
Sampling	Cake Soaps, Flake and Powdered Soap Products when Packed in Cans or Cartons	3
	Flake and Powdered Soap Products when in Bulk	4
	Liquid Soap	5
	Paste Soap Products	6
Preparation of Samples		7
Moisture	Method A. Matter Volatile at 105 C. (Oven Method)	10
	Method B. Distillation Method	11 to 13
Total Matter Insoluble in Alcohol		14 and 15
Free Alkali or Free Acid		16
Matter Insoluble in Water		17
Total Alkalinity of Matter Insoluble in Alcohol, Alkaline Salts		18
Combined Alkali, Total Anhydrous Soap		19
Chlorides		20 and 21
Unsaponified plus Unsaponifiable Matter		22 to 24
Unsaponifiable Matter		25 to 27
Unsaponified Matter		28
Rosin (Wolff's Method Modified)		29
Preparation of Total Fatty Matter, Fatty and Rosin Acids, and Unsaponified Matter		30 and 31
Titer Test		32 and 33
Acid Number of Fatty Acids		34
Borax		35 and 36
Silica Present as Alkaline Silicates		37
Carbon Dioxide (Carbonates)		38 to 40
Phosphates		41 to 43
Sulfates		44
Glycerol, Sugar and Starch	Glycerol in the Absence of Sugar	45 and 46
	Glycerol in the Presence of Sugars	47
	Starch	48
	Sugar	49 and 50
Volatile Hydrocarbons		51 to 53

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-12 on Soaps and Detergents.

SAMPLING

2. The seller shall have the option of being represented at the time of sampling and when he so requests shall be furnished with a duplicate sample.

Cake Soaps, Flake, and Powdered Soap Products when Packed in Cans or Cartons

3. One cake (can or carton) shall be taken at random from not less than 1 per cent of the seller's shipping containers, provided each package contains not less than 50 lb. (22.7 kg.). In the case of smaller containers, a cake (can or carton) shall be taken at random from each lot of containers totaling not more than 5000 lb. (2270 kg.), or fraction thereof. The gross sample shall in all cases consist of not less than three cakes (cans or cartons) taken at random from separate containers. In the case of very large lots where the sample drawn as above will amount to more than 20 lb. (9 kg.), the percentage of packages sampled shall be reduced so that the amount drawn shall not exceed 20 lb. (9 kg.). The individual cakes (cans or cartons) shall be tightly wrapped at once in paraffined paper and sealed by rubbing the edges with a heated iron. The inspector shall accurately weigh each wrapped cake (can or carton), and record its weight and the date of weighing on the wrapper. The wrapped cakes (cans or cartons) shall be placed in an air-tight container, which should be nearly filled, and which shall then be sealed, marked, and sent to the laboratory for test. Samples shall be kept cool until tested.

Flake and Powdered Soap Products when in Bulk

4. A grab sample of not less than 0.5 lb. (227 g.) shall be taken at random from not less than 1 per cent of the seller's shipping containers, provided each package contains not less than 100 lb. (45 kg.). In the case of smaller containers, a grab sample of not less than 0.5 lb. (227 g.) shall be taken at random from each lot of containers totaling not more than 10,000 lb. (4540 kg.), or fraction thereof. The gross sample shall in all cases consist of not less than three grab samples of 0.5 lb. (227 g.) each taken at random from separate containers. In the case of very large lots where the sample drawn as above will amount to more than 20 lb. (9 kg.), the percentage of packages sampled shall be reduced so that the amount drawn shall not exceed 20 lb. (9 kg.). The inspector shall rapidly mix the gross sample, place it in an air-tight container, which shall be filled, sealed, marked, accurately weighed, its weight and the date of weighing recorded on the package, and sent to the laboratory for test. Samples shall be kept cool until tested.

Liquid Soap

5. A sample of not less than 0.5 pt. (236.6 ml.) shall be taken at random from not less than 1 per cent of the seller's shipping containers, provided

each package contains not less than 10 gal. (almost 38 liters). In the case of smaller containers, a sample of not less than 0.5 pt. (236.6 ml.) shall be taken at random from each lot of containers totaling not more than 1000 gal. (37,853 liters), or fraction thereof. The gross sample shall in all cases consist of not less than three samples of 0.5 pt. (236.6 ml.) each taken at random from separate containers. Before drawing the sample from the container selected, the contents of the container shall be thoroughly agitated. The inspector shall thoroughly mix the gross sample, place it in clean, dry cans or bottles, which shall be completely filled and securely stoppered with clean corks or caps, then sealed, marked, and sent to the laboratory for test.

Paste Soap Products

6. (*a*) *When Packed in Cans or Cartons of 5 lb. (2.27 kg.) or Less.*—One can or carton shall be taken at random from not less than 1 per cent of the seller's shipping containers, provided each package contains not less than 50 lb. (22.7 kg.). In the case of smaller containers, a can or carton shall be taken at random from each lot of containers totaling not more than 5000 lb. (2270 kg.), or fraction thereof. The gross sample shall in all cases consist of not less than three cans or cartons taken at random from separate containers. In the case of very large lots where the sample drawn as above will amount to more than 20 lb. (9 kg.), the percentage of packages sampled shall be reduced so that the amount drawn shall not exceed 20 lb. (9 kg.). The samples shall be wrapped, sealed, marked, and sent to the laboratory for test.

(*b*) *When Packed in Bulk.*—A "trier" sample[1] of not less than 0.5 lb. (227 g.) shall be taken at random from not less than 1 per cent of the seller's shipping containers, provided each package contains not less than 50 lb. (22.7 kg.). In the case of smaller containers, a "trier" sample[1] of not less than 0.5 lb. (227 g.) shall be taken at random from each lot of containers totaling not more than 5000 lb. (2270 kg.), or fraction thereof. The gross sample shall in all cases consist of not less than three 0.5-lb. (227-g.) samples, each taken at random from separate containers. With very large lots where the sample drawn as above will amount to more than 10 lb. (4.5 kg.), the percentage of packages sampled shall be reduced so that the amount drawn shall not exceed 10 lb. (4.5 kg.). The inspector shall promptly place the gross sample in a clean, dry, air-tight and water-tight container, which shall be filled, sealed, marked, and sent to the laboratory for test.

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[1] A trier sample is obtained by inserting a "trier" into the material. A trier is a half-round steel cylinder ½ to ¾ in. in diameter, 6 to 36 in. in length, pointed on one end and having a grip handle on the other end. After insertion, the trier is turned two or three times and upon removal a core of the material being sampled will be obtained.

PREPARATION OF SAMPLES

7. (*a*) *Cake Soap.*—In the case of samples of cake soap that can be easily disintegrated and mixed, run the entire sample through a suitable chopper. With samples that cannot be handled in this manner, select a cake of average weight, quarter it by cutting at right angles in the center, and shave equally from all freshly cut surfaces sufficient soap for analysis. Mix the sample and weigh at once all portions for analysis, preserving the remainder in an air-tight container in a cool place.

(*b*) *Powdered and Chip Soaps.*—Rapidly disintegrate and mix the sample of powdered, flake or chip soap; if desired, quarter down to about 1 lb. (453.6 g.). Weigh at once all portions for analysis, preserving the remainder in an air-tight container in a cool place.

(*c*) *Liquid Soap.*—No preparation of the sample of liquid soap, other than a thorough mixing, is necessary unless it is received during very cold weather, when it should be allowed to stand at least 1 hr. after it has warmed up to room temperature (20 to 30 C.) before it is tested, particularly for its lathering qualities.

(*d*) *Paste Soap Products.*—Mix the sample of paste soap products thoroughly by kneading and quarter down to about 1 lb. (453.6 g.). Weigh at once all portions for analysis, preserving the remainder in an air-tight container in a cool place.

METHODS OF CHEMICAL ANALYSIS

Duplicate Tests

8. When a determination shows nonconformity with the specifications a duplicate test shall be made.

MOISTURE

9. The oven method described in Section 10 is generally applicable to all soaps, but certain exceptions to this method must be made if accurate results are desired. These exceptions include the following:

(*a*) For soaps containing appreciable amounts of sodium silicate, the distillation method (Sections 11 to 13) is preferred.

(*b*) Soaps of linseed and other oxidizing oils absorb oxygen and if the oven method is used may gain weight near the end of the test. Therefore, either an inert atmosphere or vacuum oven should be used. The distillation method is also applicable to these types of soap.

Method A. Matter Volatile at 105 C. (Oven Method)

Procedure

10. Weigh 5 g. ±0.01 g. of the sample in a porcelain or glass dish about 6 to 8 cm. in diameter and about 2 to 4 cm. in depth, and dry to constant weight in an air oven at a temperature of 105 C. ±2 C.

Method B. Distillation Method

Apparatus

11. The distillation apparatus[1] consists of the following: a 500-ml. flask of either the short-neck, round-bottom type or the Erlenmeyer type, a Liebig condenser, 19 in. in length, and a receiving tube or trap graduated in 0.1 ml., in sizes ranging from 5 ml. to 10 ml. in capacity. The trap has a side arm intake which connects to the 500-ml. flask. The traps may be procured as separate units, or as an integral part of a specialized apparatus fitted together with ground glass joints. A 5-ml. trap is required for this test. A satisfactory assembly of the apparatus is illustrated in Fig. 1.

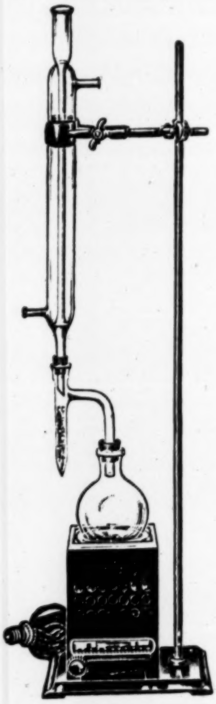

FIG. 1.—Assembly of Distillation Apparatus.

Special Solutions Required

12. *Xylol.*—Saturate the xylol with water by shaking with a small quantity of water and distill. Use the distillate for the determination.

Procedure

13. For soaps containing from 5 to 25 per cent moisture and volatile matter use 20 g. ± 0.04 g. of the sample. For soaps containing more than

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[1] The Bidwell-Sterling, and the Stark and Dean apparatus have been found satisfactory for this purpose.

25 per cent moisture and volatile matter use 10 g. ± 0.02 g. of the sample. Carefully transfer the weighed sample to the 500-ml. flask. Add approximately 10 g. of anhydrous, fused sodium acetate to prevent violent frothing, and then follow with 100 ml. of xylol which has been saturated with water. Attach the flask to a 5 to 6-ml. distilling tube receiver, calibrated at 25 C., which is connected to a 48-cm. reflux condenser (19-in. Liebig condenser). Prior to starting the determination, fill the receiver with saturated xylol by pouring in through the reflux condenser.

So that the refluxing will be under better control, wrap the flask and tube leading to the receiver with asbestos cloth. Apply heat to the flask by means of a gas burner or an electric heater and distill slowly. The rate at the start should be approximately 100 drops per minute. When the greater part of the water has distilled over, increase the distillation rate to 200 drops per minute until no more water is collected. Purge the reflux condenser during the distillation with 5-ml. portions of xylol to wash down any moisture adhering to the walls of the condenser. The water in the receiver may be made to separate from the xylol by using a spiral copper wire. Move the wire up and down in the condenser occasionally, thus causing the water to settle at the bottom of the receiver. Reflux for at least 2 hr., and shut off the heat at the end of this period. Adjust the temperature of the distillate to 25 C. Read the volume of water and calculate the percentage of moisture in the soap, as follows:

$$\text{Moisture, per cent} = \frac{\text{Volume in milliliters at 25 C.} \times 0.997}{\text{Weight of sample in grams}} \times 100$$

TOTAL MATTER INSOLUBLE IN ALCOHOL, AND FREE ALKALI OR FREE ACID

Matter Insoluble in Alcohol

Special Solutions Required

14. *Ethyl Alcohol (94 per cent).*—Freshly boiled ethyl alcohol, 94 per cent or higher, and neutral to phenolphthalein.

Procedure

15. Digest 2 to 10 g. ± 0.01 g. of the sample with 200 ml. of freshly boiled ethyl alcohol in a covered vessel on a steam bath until the soap is dissolved. Filter through a counterpoised filter paper, neutral to phenolphthalein, or through a weighed Gooch crucible with suction, protecting the solution from carbon dioxide and other acid fumes during the operation by covering with a watch glass. Wash the residue on the paper, or in the crucible, with hot neutral alcohol until free from soap and reserve the filtrate and washings. Dry the filter paper or crucible with the residue at 100 to 105 C. for 3 hr., cool, and weigh the total matter insoluble in alcohol.

NOTE.—The matter insoluble in alcohol will contain most of the alkaline salts, such as carbonates, borates, silicates, phosphates, and sulfates, as well as starch, and may be used for the approximate determination of these constituents. These salts are not entirely insoluble in alcohol, so for accurate determinations separate portions of the soap should be used. For determination of carbonates, see Sections 38 to 40; phosphates, Sections 41 to 43; sulfates, Section 44; silicates, Section 37; borax, Sections 35 and 36; and starch, Section 48.

Free Alkali or Free Acid

Procedure

16. Heat the reserved filtrate (Section 15) to incipient boiling and titrate with standard acid or alkali solution using phenolphthalein as indicator, and calculate to sodium hydroxide (or potassium hydroxide) if alkaline, or to oleic acid, if acid.

MATTER INSOLUBLE IN WATER

Procedure

17. Proceed as in the determination of matter insoluble in alcohol (Section 15). After filtering and thoroughly washing the residue, change the receivers and extract the residue with water at 60 C. and wash the filter thoroughly. (When the matter insoluble is all inorganic, boiling water may be used for the extraction and washing.) Reserve the water solution. Dry the filter and residue at 100 to 105 C. for 3 hr., cool, and weigh the matter insoluble in water. The nature of this matter may be determined by further examination.

TOTAL ALKALINITY OF MATTER INSOLUBLE IN ALCOHOL, ALKALINE SALTS

Procedure

18. Titrate the water solution obtained in the determination of matter insoluble in water (Section 17) with standard acid, using methyl orange as indicator. Calculate the alkalinity to sodium oxide (Na_2O), and, if desired, to any other basis agreed upon by the purchaser and the seller.

COMBINED ALKALI, TOTAL ANHYDROUS SOAP

Procedure

19. (*a*) Dissolve 5 to 10 g. ± 0.01 g. of the sample, depending upon the anhydrous soap content, in 100 ml. of water in a 250-ml. Erlenmeyer flask. When the solution is complete, add diluted H_2SO_4 in slight excess, insert a small funnel in the neck of the flask, and heat the flask to a temperature not exceeding 60 C. until the fatty acids separate as a clear layer. Transfer to a separatory funnel, draw off the acid layer into a second separatory funnel, and shake the acid aqueous liquid with two 20-ml. portions of ethyl ether. Dissolve the fatty acids in the ether used for washing the aqueous liquid and shake with 10-ml. portions of water until they are no

longer acid to methyl orange. Unite the water portions used for washing and shake with 20 ml. of ether. Wash this ether until the wash water is neutral to methyl orange. Reserve the acid water for the determination of chlorides (Sections 20 and 21).

(*b*) Unite the ether solutions (if necessary, filter, washing the paper with ether) in a suitable weighed vessel, add 100 ml. of neutral alcohol free from carbon dioxide, add phenolphthalein, and titrate to exact neutrality with standard 0.5 *N* NaOH solution. Evaporate the alcohol, dry to constant weight as in the determination of matter volatile at 105 C. (Section 10), and calculate the percentage of soda soap. This naturally includes any mineral oil and neutral fat which, if determined separately, must be deducted from the result to obtain the true soap. Calculate the combined sodium oxide (Na_2O) and deduct from the weight of soda soap to give the acid anhydrides. If the original soap was potash soap, proper calculation must be made to reduce to potassium oxide (K_2O), or the titration made directly with standard 0.5 *N* KOH solution. In case the soap shows an excess of free acid, proper corrections must be made in calculating the combined alkali in the original soap. (A blank test should be made on the NaOH or KOH solution for neutral salts and the proper corrections made if necessary.)

(*c*) In the case of soaps containing a large amount of soluble silicates, and soap products containing a high percentage of finely divided material insoluble in water, the foregoing procedure cannot be applied as given. In such cases the filtrate obtained in the determination of total matter insoluble in alcohol (Section 15) may be used after neutralizing any free acid or alkali. Evaporate the alcohol on a steam bath, take up with water, and proceed according to the procedure given in Paragraphs (*a*) and (*b*).

(*d*) In the case of soap products containing a high percentage of matter insoluble in alcohol where approximate results will suffice, such as cleansers, soap powders, scouring compounds, pastes, etc., and where agreed upon by the purchaser and the seller, the alcoholic solution, obtained after filtering off and washing the matter insoluble in alcohol (Section 15), may be evaporated directly in a weighed vessel, dried at 105 C. to constant weight and the result reported as soap.

CHLORIDES

Special Solutions Required

20. (*a*) *Silver Nitrate Solution (0.1 N).*

(*b*) *Potassium Chromate Indicator.*—Dissolve 5 g. of potassium chromate in water and add solution of silver nitrate until a slight red precipitate is produced, filter the solution, and dilute to 100 ml.

Procedure

21. (*a*) Neutralize with chloride-free alkali the acid water obtained in the determination of combined alkali (Section 19). Titrate with standard silver nitrate solution, using potassium chromate as indicator and calculate the result to NaCl or KCl as the character of the soap indicates.

(*b*) In case the total anhydrous soap is not to be determined, it will be more convenient to use the following method: Dissolve 5 g. ± 0.01 g. of the sample in 300 ml. of water, boiling if necessary to effect solution. Add an excess of neutral, chloride-free magnesium nitrate solution (about 25 ml. of a 20 per cent $Mg(NO_3)_2 \cdot 6H_2O$ solution). Without cooling or filtering, titrate with standard silver nitrate solution, using potassium chromate as indicator. One milliliter of the 0.1 N $AgNO_3$ solution is equivalent to 0.00585 g. of NaCl.

UNSAPONIFIED PLUS UNSAPONIFIABLE MATTER

Apparatus

22. *Extraction Cylinder.*—The extraction cylinder shall be a 250-ml. graduated glass-stoppered cylinder about 35 mm. (1.375 in.) in diameter and 30 cm. (12 in.) in length.

Special Solutions Required

23. (*a*) *Ethyl Alcohol (50 per cent).*

(*b*) *Ethyl Alcohol (95 per cent).*

(*c*) *Ethyl Alcohol (10 per cent).*

(*d*) *Sodium Hydroxide Solution (0.1 N).*

(*e*) *Sodium Hydroxide Solution (0.04 N).*

(*f*) *Petroleum Ether.*—The solvent used shall be of the pentane type, containing a minimum amount of *iso*pentane, *iso*hexane and hexane, conforming to the following requirements:

Distillation Test:	
Initial boiling point	not less than 35 C. nor more than 40 C.
Dry flask endpoint	not less than 50 C. nor more than 60 C.
Distilling under 55 C	not less than 95 per cent
Distilling under 40 C	not more than 85 per cent
Specific gravity at 15.5/15.5 C. (60/60 F.)	0.630 to 0.675
Color	water white
Doctor test	sweet
Evaporation residue	not over 0.002 per cent by weight
Copper-strip corrosion test	non-corrosive
Unsaturated compounds	trace only permitted

Procedure

24. (*a*) Weigh 5 g. ± 0.2 g. of the sample and place it in a 250-ml. Erlenmeyer flask or beaker which contains approximately 0.1 g. of bicarbonate of soda, and dissolve in 100 ml. of redistilled 50 per cent ethyl alcohol. Warm and shake to effect solution, keeping the temperature

under 60 C., and filter off any undissolved residue on a Gooch crucible with an asbestos pad or in a funnel, using an asbestos pad deposited on a perforated porcelain disk. Wash three times with hot 50 per cent alcohol. Wash with a small amount of petroleum ether to remove any traces of unsaponified and unsaponifiable matter. Transfer the entire alcohol-water-and-ether filtrate to the extraction cylinder and make up to the 160-ml. mark with redistilled, 50 per cent ethyl alcohol. Add 50 ml. of petroleum ether, shake vigorously for 1 min. (Note 1), and allow to settle until both layers are clear. The volume of the upper layer should be about 40 ml. Draw off the petroleum ether layer as closely as possible, by means of a slender glass siphon, into a separatory funnel of 500-ml. capacity.

(*b*) Repeat the extraction at least six times using 50 ml. of petroleum ether each time (Note 2). Wash the combined ether extracts first with a mixture of 15 ml. of 0.1 *N* sodium hydroxide solution and 15 ml. of 95 per cent alcohol, and then three times with 25-ml. portions of 10 per cent alcohol, shaking vigorously each time. Transfer the petroleum ether extract to a beaker and evaporate the petroleum ether on a steam bath by the aid of a current of air.

NOTE 1.—Thorough and vigorous shaking is necessary in order to secure accurate results. The two phases must be brought into the most intimate contact possible; otherwise low and disagreeing results may be obtained.

NOTE 2.—This method will not remove all the unsaponifiable matter in soaps to which lanolin has been added. More extractions are required when substances of this nature are present.

(*c*) Test the residue for solubility by treating with 50 ml. of petroleum ether at room temperature. Filter, and wash free from the insoluble residue, if any; evaporate and dry in the same manner on a steam bath, and finally in an air oven at 100 to 101 C. for 30 min. Weigh, and return to the oven, reweighing at 15-min. intervals until constant weight is reached. Take up the residue in 50 ml. of warm ethyl alcohol, neutralized to phenolphthalein, titrate to the same color as the original neutral alcohol with 0.04 *N* NaOH solution and calculate to oleic acid. Deduct this figure from the gross weight previously found and report as "unsaponified and unsaponifiable matter."

(*d*) Make a blank test on the petroleum ether by evaporating 250 ml. of the ether with about 0.25 g. of stearin or some other hard fat previously brought to constant weight by heating and drying as in the actual determination. The blank must not exceed a few milligrams.

NOTE.—Any blank from the petroleum ether must be deducted from the weight before calculating the unsaponified and unsaponifiable matter.

UNSAPONIFIABLE MATTER

Apparatus

25. *Extraction Cylinder.*—See cylinder described in the determination of unsaponified plus unsaponifiable matter (Section 22).

Special Solutions Required

26. (*a*) See the special solutions required in the determination of unsaponified plus unsaponifiable matter (Section 23).

(*b*) *Potassium Hydroxide Solution* (*50 per cent*).

Procedure

27. Weigh 5 g. ± 0.2 g. of the sample and place in a 200-ml. Erlenmeyer flask. Add 30 ml. of redistilled 95 per cent alcohol and 5 ml. of aqueous KOH (50 per cent), and boil for 1 hr. under a reflux condenser. Transfer to the extraction cylinder and wash to the 40-ml. mark with redistilled 95 per cent ethyl alcohol. Complete the transfer, first with warm and then with cold water, until the total volume is 80 ml., and finally with a small quantity of petroleum ether. Cool the cylinder to room temperature and add 50 ml. of petroleum ether; then proceed with the extraction as outlined in the procedure for unsaponified plus unsaponifiable matter (Section 24; see also Notes 1 and 2), except that the alkaline wash may be omitted. Weigh the residue and correct for fatty acids in the usual manner. Report the result as unsaponifiable matter.

UNSAPONIFIED MATTER

28. From the total unsaponified plus unsaponifiable matter determined in Section 24, deduct the unsaponifiable figure obtained in Section 27 and report the difference as "unsaponified matter."

Rosin (Wolff's Method, Modified)

Procedure

29. (*a*) *Preparation of Fatty and Rosin Acids.*—Dissolve 5 g. ± 0.01 g. of the sample in 100 to 200 ml. of hot distilled water in a 250-ml. beaker, add a slight excess of dilute sulfuric acid (1:1), heat on a steam bath until the fatty acids collect in a clear layer, cool to room temperature, and transfer to a separatory funnel, washing the beaker free from fatty and rosin acids with small portions of ethyl ether, adding the ether washings to the separatory funnel, designated as No. 1. Add about 50 ml. of ethyl ether to the separatory funnel and whirl to dissolve the fatty and rosin acids. After the solid acids have dissolved in the ether, allow the solution to stand for at least 5 min.

Draw off the aqueous portion into another separatory funnel designated as No. 2, add about 30 ml. of ethyl ether to funnel No. 2, shake vigorously, allow to stand for at least 5 min., and then draw off the aqueous portion into another separatory funnel designated as No. 3. Transfer the ether extract to separatory funnel No. 1. Wash the aqueous liquid three more times, using for each washing about 30 ml. of ethyl ether, shaking vigorously, then discard the aqueous liquid and add the ether washings to funnel No. 1. Now add 50 ml. of distilled water to the combined ether extracts in funnel No. 1, shake vigorously, and allow to stand for 5 min. Draw off the aqueous solution into another separatory funnel. Repeat this operation, combining the water washings until the ether is free from acid, using methyl orange as indicator. Add 50 ml. of ethyl ether to the water washings, shake the separatory funnel vigorously, and allow to stand for at least 5 min. Draw off and discard the aqueous solution and wash the ether layer with small portions of distilled water until free of acid, using methyl orange as indicator. Transfer the total ether extract to a 250-ml. Erlenmeyer flask, evaporate the ether slowly on the side of a steam bath, dry 1 hr. in an oven at 105 C., cool, and dissolve in 20 ml. of absolute alcohol.

(*b*) *First Esterification.*—Add 10 ml. of a solution of one volume of H_2SO_4 (sp. gr. 1.84) in four volumes of absolute ethyl alcohol, bring to a boil on the steam bath, and boil exactly 4 min. under a reflux condenser. Remove from the steam bath, add to the liquid about five times its volume of a NaCl solution (7 to 10 per cent). Transfer to a separatory funnel, washing the Erlenmeyer flask with small portions of ethyl ether, the ether washings being added to the aqueous solution in the separatory funnel. Add about 30 ml. of ethyl ether to the separatory funnel No. 1, shake vigorously, and allow to stand for at least 5 min.

Draw off the aqueous solution into another funnel, No. 2. Add about 30 ml. of ether to funnel No. 2, shake vigorously, allow to stand for at least 5 min., and then draw off the aqueous layer into another separatory funnel, No. 3. Repeat the extraction of the aqueous solution three times more. Then discard the aqueous liquid and add the combined ether solutions to separatory funnel No. 1. Add 50 ml. of NaCl solution (7 to 10 per cent) to the combined ether extracts, shake vigorously and allow to stand for at least 5 min. Draw off the aqueous solution into another separatory funnel. Repeat this operation until the washings are neutral to methyl orange, all salt water washings being combined. Add 50 ml. of ether to the washings in the separatory funnel. Shake the separatory funnel vigorously, allow to stand for at least 5 min., and then draw off and discard the aqueous salt solution. Add 50 ml. of NaCl solution and wash the ether layer until the aqueous salt solution is neutral to methyl orange. Dry the ether extract by the addition of about 3 g. of anhydrous Na_2SO_4 to the separatory funnel, filter into a 250-ml. Erlenmeyer flask and evaporate off the ether slowly on a steam bath to dryness.

(*c*) *Second Esterification.*—Cool, and dissolve the residue in 20 ml. of absolute ethyl alcohol and then proceed as described for the first esterification in Paragraph (*b*).

(*d*) Add 30 ml. of neutral alcohol (94 per cent or higher) and titrate rosin or rosin soap as desired, using phenolphthalein as indicator (1 ml. 0.5 N alkali = 0.173 g. of rosin or 0.188 g. of rosin soda soap). If the pure fatty acid soap is desired, subtract the rosin soap from the total anhydrous soap obtained in Section 19.

Note.—This method gives somewhat high results with low-rosin soap and approximately correct results with high-rosin soap. In all cases where the rosin content is found to be less than 5 per cent, the actual presence or absence of rosin should be checked qualitatively by the Liebermann-Storch test, which is as follows:

Transfer 1 to 2 ml. of the sample of fatty acids to a test tube, treat with 5 to 10 ml. of reagent grade acetic anhydride, and warm on a steam bath. After cooling, pour 1 to 2 ml. into a white porcelain dish and allow a drop or two of sulfuric acid (sp. gr. 1.53) to run down the side of the vessel. (The sulfuric acid is prepared by diluting 34.7 ml. of H_2SO_4 (sp. gr. 1.84) with 35.7 ml. of distilled water.) If rosin is present, a fugitive violet coloration changing to a brownish tinge is immediately produced at the margin of contact of the reagents. The test should be checked with a sample of fatty acids to which a small amount of rosin has been added.

PREPARATION OF TOTAL FATTY MATTER

(FATTY AND ROSIN ACIDS, AND UNSAPONIFIED MATTER)

Special Solutions Required

30. *Sulfuric Acid (30 per cent).*—Slowly add 650 g. of H_2SO_4 (sp. gr. 1.84) to 1400 ml. of water.

Procedure

31. (*a*) *Preparation for Titer Test Only.*—Dissolve about 50 g. of the sample in 500 ml. of hot water, add 100 ml. of H_2SO_4 (30 per cent), heat until the fatty matter collects in a clear layer. Siphon off the aqueous acid layer and wash the fatty matter free from H_2SO_4 with hot water. Decant the fatty matter into a dry beaker, filter, using a hot-water funnel, or placing both funnel and receiving beaker in a water-jacketed oven, and dry for 20 min. at 100 C.

(*b*) *Preparation for Other Tests.*—When other determinations are to be made on the total fatty matter, and volatile and readily oxidizable fatty acids are present, the following method should be used: Dissolve about 50 g. of the sample in 300 ml. of hot water, and transfer to a separatory funnel. Add 150 ml. of approximately 2 *N* H_2SO_4 to the solution in the funnel, cool somewhat and add 120 ml. of ether. Shake the solution, draw off the acid layer, and wash the ether layer free from acid with a strong NaCl solution. Draw off the aqueous layer as completely as possible, transfer the ether layer to a flask (it is not necessary to transfer quantitatively), add 20 to 30 g. of anhydrous Na_2SO_4, stopper the flask, shake and allow to stand at a temperature below 25 C. untll the ethereal liquid becomes perfectly clear, showing that all the water had been taken up by the Na_2SO_4. Filter through a dry filter paper into an Erlenmeyer flask, and completely evaporate the ether by passing through the flask a current of dry air and heating the flask to a temperature of about 50 C.

TITER TEST

Apparatus

32. *Thermometer.*—The thermometer shall be graduated at zero in 0.1 deg. from 10 C. to 65 C. and have an auxiliary reservoir at the upper end. The 10 C. mark shall be about 3 to 4 mm. above the bulb and the length of the thermometer shall be about 37 cm. over all. The thermometer shall have been annealed for 75 hr. at 450 C. and the bulb shall be normal 16 in. glass[1] or its equivalent, moderately thin so that the thermometer will be quick acting. The bulb shall be about 3 cm. in length and 6 mm. in diameter. The stem of the thermometer shall be 6 mm. in diameter and made of the best thermometer tubing, with the scale etched on the stem, the graduation being distinct but quite fine. The thermometer shall be certified by the National Bureau of Standards.

Procedure

33. Transfer the fatty acids (Section 31 (*a*)) when cooled somewhat, to a titer tube 25 by 100 mm. placed in a 273 to 275-ml. (16-oz.) saltmouth bottle of clear glass 70 by 150 mm., fitted with a cork that is perforated so as to hold the tube rigidly when in position. Suspend the titer thermometer so that it can be used as a stirrer and stir the fatty acids slowly (with a circular motion in one plane at about 100 r.p.m.) until the mercury remains stationary for 30 sec. Allow the thermometer to hang quietly with the bulb in the center of the tube and report the highest point to which the mercury rises as the titer of the fatty acids. The titer should be made in a room at about 20 C. for all fats having a titer above 30 C. and at 10 C. below the titer for all other fats.

ACID NUMBER OF FATTY ACIDS

Procedure

34. For the preparation of the sample for this determination follow the procedure described in Section 31 (*b*). In a 250-ml. Erlenmeyer flask dissolve 2 g. of the fatty acids, accurately weighed, in 20 to 30 ml. of neutral 95 per cent ethyl alcohol. Titrate with standard alkali, using phenolphthalein as indicator. Calculate the acid number (milligram of KOH per gram of fatty acids).

BORAX

Special Solutions and Materials Required

35. (*a*) *Sodium Hydroxide Solution* (*0.1 N*).

(*b*) *Silica, Fine Powder.*

(*c*) *Precipitated Calcium Carbonate.*

(*d*) *Glycerol.*

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[1] Jena glass is very satisfactory for this purpose.

Procedure

36. (*a*) Weigh 10 g. ± 0.02 g. of the sample (or 5 g. ± 0.01 g. if more than 5 per cent of borax is present) into a platinum dish and add 2.15 g. of fusion mixture consisting of 200 g. of sodium carbonate, 15 g. of silica in fine powder. To this mixture add 15 ml. of alcohol (94 per cent or higher), mix with the aid of a glass rod, and after washing the rod with a little alcohol, evaporate the mass to dryness on a water bath. Ignite until the combustible material is destroyed, cover the dish with a piece of platinum foil and fuse.

(*b*) Completely disintegrate the fusion by boiling with water and transfer the solution to a 250-ml. round-bottom flask. Acidify the mixture with 20 ml. of diluted HCl (1:1), heat nearly to boiling, and add a moderate excess of dry precipitated calcium carbonate. Connect the flask with a reflux condenser and boil vigorously for 10 min. Filter out the precipitate through a folded filter, washing several times with hot water, but keeping the total volume of the liquid below 100 ml.

(*c*) Return the filtrate to the flask, add a pinch of calcium carbonate and again boil under a reflux condenser. Remove the flame and connect the top of the condenser with a water pump; apply suction until the boiling has nearly ceased. Cool the mixture to room temperature, add 50 ml. of neutral c. p. glycerol, and titrate the solution with 0.1 *N* NaOH free from carbonate, using phenolphthalein as indicator. After the end point is reached, add 10 ml. more of glycerol and again titrate. Repeat this process until the addition of glycerol causes no further action on the end point. The number of milliliters of NaOH required for the titration, multiplied by 0.00955 will give the equivalent of borax ($Na_2B_4O_7 \cdot 10H_2O$) present in the solution.

SILICA PRESENT AS ALKALINE SILICATES

Procedure

37. (*a*) When the material contains no mineral matter that is insoluble in water, ignite a sample of the soap containing not to exceed 0.2 g. of silica in a platinum dish at a low temperature. When charred, extract the soluble salts with water, return the paper and charred residue to the dish and complete the ignition. Unite the residue in the dish and the water extract, carefully acidify with HCl, finally adding the equivalent of from 5 to 10 ml. of strong HCl in excess. The dish or casserole containing the solution should be covered with a watch glass while adding acid so as to avoid loss by spray.

(*b*) When the material contains mineral matter insoluble in water, or a determination of highest accuracy is not necessary, take a portion of the solution after titrating the matter insoluble in alcohol (Section 18) containing not more than 0.2 g. silica and add 5 to 10 ml. of strong HCl.

(c) Evaporate the acidified solution (washing off and removing the cover glass if used) to dryness on a steam bath or hot plate at a temperature not exceeding 200 C. Cool, moisten with HCl (sp. gr. 1.19), let stand 5 to 10 min., breaking up all lumps with a stirring rod. Add about 25 ml. of hot water. Heat a few minutes and filter through a small ashless paper. Wash thoroughly with hot water.

(d) Evaporate the filtrate to dryness and repeat the above treatment, filtering on a second paper. Carefully ignite the two papers and contents in a weighed platinum crucible, first at a low temperature until the paper is consumed, then over a blast lamp. Cool in a desiccator, weigh and repeat until constant weight is obtained. If extreme accuracy is desired, moisten the weighed contents of the crucible with water, add 10 ml. of HF and four drops of strong H_2SO_4, evaporate to dryness over a low flame, ignite at the temperature of the blast lamp for about 2 min., cool in a desiccator, and weigh. The difference between this weight and the previous weight is the weight of the silica (SiO_2). To calculate sodium silicate having the ratio, 1 Na_2O:3.25 SiO_2, multiply the weight of SiO_2 by 1.308.

CARBON DIOXIDE (CARBONATES)

38. For most determinations the dry matter insoluble in alcohol and soluble in water as obtained in the determination of matter insoluble in alcohol (Section 15) will be suitable for this determination and the carbonates can be determined by direct titration of the water solution. In some cases it might be desired to run the test directly on an original sample of the soap. This should always be done when the highest accuracy is required. Any reliable absorption method for determining carbon dioxide may be used. A method which has proved satisfactory is described in the following Sections 39 and 40.

Apparatus Assembly

39. Place a 250-ml. Erlenmeyer flask on a gauze over a burner. Fit the flask with a two-hole rubber stopper, one opening to carry a 25-cm. (10-in.) reflux condenser and the other a thistle tube equipped with a three way stopcock. Draw out the end of the thistle tube to a small point, and place it in the stopper so that the point is very close to the bottom of the flask. Attach a small funnel to the straightaway end of the stopcock for the introduction of acid into the flask. Attach the other opening of the stopcock (which is to receive air) to a purifying tube containing soda-asbestos (ascarite) or any other suitable carbon dioxide absorbent. Attach to the top of the reflux condenser a train consisting of the following: (a) a drying tube containing a dehydrating agent such as sulfuric acid (sp. gr. 1.84) or magnesium perchlorate, (b) a weighed tube containing ascarite

and magnesium perchlorate, and a second weighed tube containing concentrated sulfuric acid. Attach to this train a protective U-tube containing calcium chloride; attach the U-tube to an aspirator.

Procedure

40. Set up the apparatus, leaving out the weighed train, and aspirate with a slow stream of the dry carbon dioxide-free air until the apparatus is free of carbon dioxide. Insert the train and continue to aspirate for 30 min. Check the weight of the train to determine whether the air is passing through too fast, or whether the system is free of carbon dioxide. The system must be free from leaks. Weigh out 1 or 2 g. of the sample into the Erlenmeyer flask, cover with 20 ml. of freshly boiled distilled water and close the apparatus with the train in place. Add 20 ml. of diluted HCl (1:1) very slowly through the funnel; do not apply heat to the flask. The rate of adding acid should be carefully controlled so that the gas does not pass through the train too rapidly. As soon as the acid is added, start aspiration gently. When the absorption begins to slacken, start heating gently and continue until the contents of the flask have boiled 15 to 20 min. Stop heating and continue aspirating until the flask has cooled down. Remove the train and weigh. The increase in weight represents carbon dioxide. The amount of this increase multiplied by 2.41 equals sodium carbonate.

PHOSPHATES

41. If a qualitative test has shown the presence of phosphates and their determination is desired, the matter insoluble in alcohol (Section 15) or the ash from the incineration of an original sample may be used. An original sample should always be used when the highest accuracy is desired.

Special Solutions Required

42. (*a*) *Molybdate Solution.*—Dissolve 100 g. of molybdic acid in diluted NH_4OH (144 ml. of NH_4OH (sp. gr. 0.90) and 271 ml. of water); pour this solution slowly and with constant stirring into dilute nitric acid (489 ml. of HNO_3 (sp. gr. 1.42) and 1148 ml. of water). Keep the final mixture in a warm place for several days or until a portion heated to 40 C. deposits no yellow precipitate of ammonium phosphomolybdate. Decant the solution from any sediment and preserve in glass-stoppered vessels.

(*b*) *Ammonium Nitrate Solution.*—Dissolve 100 g. of commercial ammonium nitrate, phosphate-free, in water, and dilute to 1 liter.

(*c*) *Magnesia Mixture.*—Dissolve 55 g. of crystallized magnesium chloride ($MgCl_2 \cdot 6H_2O$) in water, add 140 g. of ammonium chloride, 130.5 ml. of NH_4OH (sp. gr. 0.90), and dilute to 1 liter.

(*d*) *Diluted Ammonium Hydroxide for Washing.*—Dilute 100 ml. of NH_4OH (sp. gr. 0.90) to 1 liter.

Procedure

43. (*a*) Weigh out a 2-g. ± 0.01 g. sample of the alcohol-insoluble matter or ash and proceed as in the determination of alkaline silicates (Section 37) for removal of silica, saving the filtrate. Make up the filtrate to 250 ml. concentrating if necessary. Pipette an aliquot corresponding to 0.50 or 1 g. into a 250-ml. beaker; add NH_4OH in slight excess; and barely dissolve the precipitate formed with a few drops of HNO_3, stirring vigorously. Add about 15 g. of dry ammonium nitrate or a solution containing that amount. Heat to about 60 C. and add 70 ml. of the molybdate solution for every decigram of phosphoric acid (P_2O_5) present.

(*b*) Digest at about 65 C. for 1 hr. and determine whether or not the phosphoric acid has been completely precipitated by the addition of more molybdate solution to the clear supernatant liquid. Filter, and wash with cold water or preferably with the ammonium nitrate solution. Dissolve the precipitate on the filter with NH_4OH (1:1) and hot water and wash into a beaker to a volume of not more than 100 ml. Neutralize with HCl using litmus paper or bromthymol blue as an indicator, cool, and from a burette add slowly (about 1 drop per second), stirring vigorously, 15 ml. of the magnesia mixture for each decigram of phosphoric acid (P_2O_5) present. After 15 min., add 12 ml. of NH_4OH (sp. gr. 0.90).

(*c*) Let the solution stand until the supernatant liquid is clear (2 hr. is usually enough), filter, wash the precipitate with the diluted NH_4OH until the washings are practically free from chlorides; dry, burn first at low heat and ignite to constant weight, preferably in an electric furnace, at 950 to 1000 C.; cool in a desiccator, and weigh as $Mg_2P_2O_7$. Calculate and report the result as percentage of P_2O_5 or alkaline phosphate known to be present.

SULFATES

Procedure

44. For most determinations the matter insoluble in alcohol obtained in Section 15 may suffice. If a determination of the highest accuracy is desired, ignite 10 g. ± 0.10 g. of the sample and use the ash from the ignition. Digest with 100 ml. of water, cover with a watch glass, and neutralize carefully with HCl. When neutralized, add 5 ml. excess of HCl, filter, and wash the residue thoroughly. (Evaporation to dryness is unnecessary unless gelatinous silica should have separated, and should never be performed on a bath heated by gas.) Make up the filtrate to 250 ml. in a beaker, and boil. To the boiling solution add 15 to 20 ml. of barium chloride solution (10 per cent) slowly drop by drop from a pipette. Continue boiling until the precipitate is well formed, or digest on a steam bath overnight. Set aside overnight or for a few hours, filter through a prepared Gooch crucible, ignite gently, and weigh as barium sulfate. Calculate to sodium sulfate, or alkaline sulfate known to be present.

GLYCEROL, SUGARS AND STARCH

Glycerol in the Absence of Sugars

Special Solutions Required

45. (*a*) *Potassium Bichromate Solution.*—Dissolve 74.553 g. of potassium bichromate in 500 ml. of water in a 1-liter volumetric flask. Dilute to the mark with water.

(*b*) *Sodium Thiosulfate Solution* (*0.1 N*).

(*c*) *Potassium Iodide Solution* (*10 per cent*).

Procedure

46. (*a*) Weigh a portion of the sample equivalent to not more than 3.0 g. of glycerol and dissolve in 200 ml. of hot water in a 600-ml. beaker. If starch is present, it will be necessary to remove the matter insoluble in water as described in Sections 15 and 17. Combine the alcohol and water solutions, evaporate off the alcohol, and proceed. Decompose with 25 ml. of H_2SO_4 (1:4). If alcohol is present, volatilize it by boiling for 20 to 30 min. Cool, remove, and rinse the cake of fatty acids, transfer the acid water and rinsings to a 500-ml. graduated flask, and add about 0.25 g. of silver sulfate to precipitate traces of chlorides and soluble fatty acids. Make up to volume and mix contents thoroughly.

(*b*) Transfer a filtered, accurately measured 50-ml. aliquot of the solution obtained in Paragraph (*a*) to a 400-ml. beaker, and to this add 75 ml. of accurately measured potassium bichromate solution, followed by 25 ml. of H_2SO_4 (sp. gr. 1.84), cover with a watch glass, and oxidize by heating to 90 to 100 C. for 3 hr. Conduct a blank in like manner but using 100 ml. of water, 25 ml. of H_2SO_4 (sp. gr. 1.84), and 25 ml. of accurately measured potassium bichromate. Cool and dilute the solutions to 1000 ml. in graduated flasks. The excess of potassium bichromate is determined by taking 50-ml. aliquots of the above, adding 50 ml. of water and 20 ml. of potassium iodide solution (10 per cent), and titrating the liberated iodine with 0.1 *N* thiosulfate, using starch solution as indicator.

(*c*) Calculate the percentage of glycerol (1 ml. of the potassium bichromate solution is equivalent to 0.0100 g. of glycerol).

Glycerol in the Presence of Sugars

Procedure

47. Proceed as in the determination of glycerol in the absence of sugar (Section 46), taking a sample so that the sum of the glycerol and sugar is not more than 3.0 g. (If starch is present, this must first be removed as described in Section 46.) The solution must be boiled in all cases at least 20 min. to ensure complete inversion of sucrose as in Section 50. Determine the amount of potassium bichromate solution required to oxidize

both the sugar and glycerol. Determine also the sugar by the method described in the determination of sugar (Sections 49 and 50).

Calculate the percentage of glycerol after deducting the amount of potassium bichromate required by the sugar.

1 ml. of potassium bichromate is equivalent to 0.0100 g. of glycerol
1 ml. of potassium bichromate is equivalent to 0.01142 g. of invert sugar

Starch

Procedure

48. (*a*) Separate the matter insoluble in water as in Section 17, using a sample of soap that will give not more than 3 g. of starch. Transfer the insoluble matter, without drying, to a beaker and heat for 2.5 hr. with 200 ml. of water and 20 ml. of HCl (sp. gr. 1.125) in a flask provided with a reflux condenser. Cool, and nearly neutralize the solution with NaOH. Make up the volume to 250 ml., filter, and determine the reducing sugars by the gravimetric method as described in the method for the determination of sugar (Section 50).

(*b*) Calculate the amount of dextrose (*d*-glucose) equivalent to the cuprous oxide obtained. This multiplied by 0.90 equals the amount of starch.

Sugars

Apparatus and Solutions Required

49. The apparatus and solutions required shall be the same as those described in the standard Munson-Walker method.[1]

Procedure

50. Dissolve 10 g. ± 0.01 g. of the sample in 200 ml. of hot water in a 600-ml. beaker. Decompose with 25 ml. of H_2SO_4 (1:4), boil gently for 20 min. to invert the sucrose completely. Cool the solution, remove the heat, and rinse the cake of fatty acids. Extract the acid liquid with 25 ml. of ether. Neutralize the acid liquid with NaOH solution, transfer to a 500-ml. graduated flask, dilute to the mark, and mix thoroughly. Determine the invert sugar in 50 ml. of this solution by the Munson-Walker method.[1] To calculate sugar (sucrose) multiply the amount of invert sugar found by 0.95. (If starch is present, first remove as described in Section 46, and then proceed as above.)

VOLATILE HYDROCARBONS

51. This method requires a source of dry, oil-free steam which is passed through the sample treated with acid, sufficient to liberate the fatty acids from the soap. The steam is next passed through strong NaOH solution to scrub out any volatile fatty acids, while the volatile hydrocarbons are

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[1] Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists, Sugar and Sugar Products, Section XXXIV, Paragraph 28, p. 476, 4th Edition (1935).

condensed with the steam in a suitable arrangement which allows the excess water to flow away, leaving the volatile hydrocarbons in the measuring burette. The method may be applied to samples containing substances immiscible with water and volatile with steam. (For solvents heavier than water a Bidwell-Sterling tube should be used. (See Sections 11 to 13.))

Apparatus

52. The apparatus and its arrangement are shown in Fig. 2. The following are the important items:

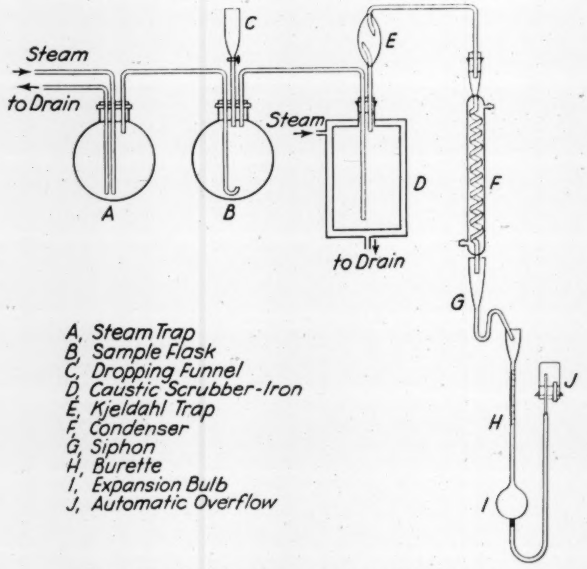
Steam
to Drain
C
E
Steam
A
B
D
F
to Drain
G
J
H
I
A, Steam Trap
B, Sample Flask
C, Dropping Funnel
D, Caustic Scrubber-Iron
E, Kjeldahl Trap
F, Condenser
G, Siphon
H, Burette
I, Expansion Bulb
J, Automatic Overflow

FIG. 2.—Volatile Hydrocarbon Apparatus.

(*a*) *Steam Trap, A*.—A 1-liter round-bottom ring-neck flask equipped with a siphon tube to the drain from the bottom of the flask and provided with a means of regulating the steam flow into the flask.

(*b*) *Evolution or Sample Flask, B*.—A 1-liter round-bottom ring-neck flask. In case large samples are desirable the size of this flask may be increased.

(*c*) *Caustic Scrubber Flask, D*.—A steam jacketed metal flask is preferred, but a 1-liter Florence flask provided with a steam coil of 0.32 cm. (0.125 in.) copper tubing around the upper half may be used. If the glass flask is used it should be provided with a safety bucket below it and should be renewed frequently since the strong caustic dissolves the glass rather rapidly. This flask should be connected to the condenser by a Kjeldahl connecting tube, *E*, or similar safety device.

The inlet for the steam into the evolution and scrubber flasks should extend nearly to the bottom of the flasks and bent at right angles and parallel to the sides of the flask.

(*d*) *Condenser, F.*—A 30.5-cm. (12-in.) or longer spiral condenser of sufficient bore so the condensate will not readily close it.

(*e*) *Measuring Burette, H.*—A 10-ml. burette calibrated to 0.1 ml. and carrying a bulb, *I*, of approximately 100-ml. capacity, at the lower end.

The stoppers used should be of a good grade of rubber and should have been thoroughly cleaned free from any surface sulfur and should be given a steam distillation in position for several hours before use on a sample.

Insulating the flasks and tubing to reduce condensation aids distillation and its control.

Procedure

53. (*a*) Place 150 ml. of NaOH solution (approx. sp. gr. 1.47) and several sticks of solid sodium hydroxide to provide against dilution in the scrubber flask. Rinse out the condenser and burette with acetone. Attach a rubber tubing to the lower end of the burette, fill the burette and tubing with water, and raise the outer end of the tubing so that the water level in the burette is near the top of the scale when the water is flowing to the drain from the automatic overflow, *J*. Be sure that the connections are tight and that the tubing contains no air bubbles. Place the condenser in position so that the lower end extends directly into the upper end of the burette just above the water level or connect to an adapter siphon, *G*, which discharges into the burette. The cooling water should be 15.5 C. or colder. Ice water may be desirable for low-boiling hydrocarbons.

(*b*) Weigh 100 g. ± 0.5 g. of the sample (cut into cubes of about 1-cm. edges) or 50 g. ± 0.3 g. of the sample of soap powder and transfer to the evolution flask (Note). Add about 10 g. of gum arabic (commercial) and 100 ml. of distilled water. Place the flask in position with 100 ml. of H_2SO_4 (1:3) in a dropping funnel, *C*, carried in the stopper. Connect with the steam and wash the flasks and the condenser, making sure that the stoppers are tightly fitting and held in place by wiring. Rubber connections in the lines between the evolution flask and condenser should be avoided.

(*c*) Add the acid to the sample slowly to avoid excessive frothing. While adding the acid, turn on the steam cautiously, so adjusting the pressure by a bleeder valve that just enough steam flows to prevent any liquid from backing into the steam trap flask.

(*d*) When all the acid has been added, turn on enough steam to cause brisk distillation, taking care that no liquid is carried over from the evolution and wash flasks and that the condenser water does not become warm.

(*e*) Continue the distillation until there is no increase in the volume of the upper layer for 45 min. or no small droplets can be noted in the condensate.

(*f*) When distillation is completed, shut off and drain the condenser water, and allow the steam to heat up the condenser to drive out the last traces of volatile hydrocarbon. Shut off the steam as soon as vapor begins to issue from the lower end of the condenser. Immediately open the stopcock of the dropping funnel to prevent NaOH being drawn into the evolution flask.

(*g*) Stopper the burette and allow its contents to come to room temperature or bring them to a definite temperature by immersing the burette for 1 to 2 hr. in a water bath held at 25 C.

(*h*) Read the volume of the upper layer to the nearest 0.01 ml. The volume multiplied by the specific gravity equals the weight of the volatile hydrocarbon. The specific gravity should be determined at the temperature at which the volume is read. A small Sprengel tube made of 3-mm. glass tubing is convenient for this purpose.

(*i*) *Calculation.*—Calculate the percentage of volatile hydrocarbons as follows:

$$\text{Volatile Hydrocarbons, per cent} = \frac{\text{Milliliters of volatile hydrocarbon} \times \text{sp. gr.}}{\text{Weight of sample in grams}} \times 100$$

NOTE.—For some samples the volatile hydrocarbon content may be so low that a larger sample than 50 or 100 g. is desirable. The size of the evolution flask may need to be increased if larger samples are used. The amount of water in the evolution flask and acid used should also be correspondingly increased.

TENTATIVE DEFINITIONS
OF
TERMS RELATING TO SOAPS AND DETERGENTS[1]

A.S.T.M. Designation: D 459 – 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1937.

Soap.—The product formed by the saponification of fats, oils, rosins, or their acids, with organic or inorganic bases.

Commercial Soap.—Soap, with or without builders.

Anhydrous Real Soap.—The product of the combination of fatty or rosin acid anhydrides and the equivalent amount of organic or inorganic basic oxides. It is free of water and all other concomitants.

Powdered Soap.—A commercial soap that has been reduced to small particle size.

Built Soap.—Soap containing one or more builders.

Soap Content.—The quantity of anhydrous real soap present.

Soap Powder.—A water-soluble powdered product consisting of soap and one or more builders, in which the soap is a minor constituent.

Alkali Cleanser.—A water-soluble product, having an alkaline reaction and detergent qualities, but containing no soap.

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[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee D-12 on Soaps and Detergents.

TENTATIVE METHOD OF TEST

FOR

SHORT-TIME HIGH-TEMPERATURE TENSION TESTS OF METALLIC MATERIALS[1]

A.S.T.M. Designation: E 21 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1933; REVISED, 1934, 1937.

Scope

1. This method covers short-time high-temperature tension tests of metals within the range from room temperature to 2000 F. In the interests of coordination of data, it is recommended that the tests be made, as far as possible, at the following temperatures: 70, 300, 500, 750, 900, 1000, 1100, 1200, 1400, 1600, 1800 and 2000 F. It is recognized that for special purposes, tests at other temperatures may be desirable.

Selection of Metals for Test

2. This method presupposes agreement upon the selection of suitable samples for test specimens to meet the requirements of individual applications.

NOTE.—In the testing of some materials it is customary and often necessary to subject the specimens to a stabilizing treatment. If such is done, it shall be reported fully in the recorded data.

Test Specimens

3. The tension test specimen shall be either of the two types shown in Fig. 1 (*a*) and (*b*). When the type shown in Fig. 1 (*b*) is used, the specimen shanks must extend beyond the furnace before gripping. The diameter of the test specimen shall be 0.505 in. ± 0.001 in. The diameter of the reduced section of the specimen between base of fillets shall not vary by more than ±0.0005 in. The surface shall be machined smooth and free from tool marks or scratches. It shall be finished by polishing with No. 00 emery cloth. The ends of the test specimen shall be threaded, shall be at least $\frac{3}{4}$ in. in diameter, and shall be

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the Joint A.S.M.E. - A.S.T.M. Research Committee on Effect of Temperature on the Properties of Metals.

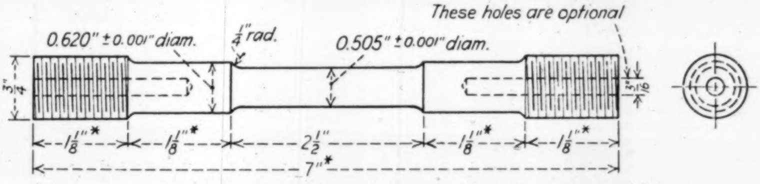
Cooperation was given by Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys of the American Society for Testing Materials in the preparation of this tentative method of test.

machined true and axial. They shall be gripped in the testing machine in such manner that the load shall be axially applied.

Testing Apparatus

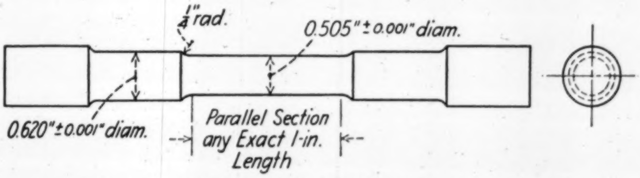
4. (*a*) *Testing Machine.*—The type of testing machine to be used in short-time high-temperature tension tests is not specified, but shall be reported with the results of a series of tests. The accuracy of the testing machine shall be within the tolerances specified in the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation: E 4) of the American Society for Testing Materials.[1]

(*b*) Precautions shall be taken to insure that the testing load is axially applied. Each specimen when set up for test shall be inspected for faulty alignment in the machine. Eccentricity of loading can often be detected by elastic extension measurements taken at room temperature before commencing the test. Apparatus provided with extensometers affording measurements on opposite sides will reveal unsatisfactory alignment when unequal strain is shown by the opposite readings. Elongation measuring devices which show extension on one side of the specimen only do not readily lend themselves to the detection of eccentric loading.



*These are preferred lengths and may be changed to suit existing conditions.

(*a*) Specimen with Threaded Ends.



(*b*) Specimen with Blank Ends.

FIG. 1.—Test Specimens for High-Temperature Tension Test.

(*c*) *Shackles.*—The type of shackles used to obtain axial loading is not specified, but a detailed description of the shackles used shall be reported with the results of a series of tests.

NOTE.—It is to be noted that eccentricity of loading of the specimen may be lessened by using rods or wire cables of the greatest length possible to connect the shackles to the heads of the testing machine.

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[1] 1936 Book of A.S.T.M. Standards, Parts I and II, pp. 849 and 1417, respectively.

Furnace and Heating

5. (*a*) A drawing showing the furnace and heating element in detail shall be submitted with the results of a series of tests.

(*b*) The design of the furnace shall be such that the maximum temperature difference between any two of the points T_i or T_o, T_f, C_o, B_f, and B_i, or B_o shown in Fig. 2 (see Form I), will not exceed 10 F. for temperatures up to and including 1600 F. and will not exceed 20 F. for higher temperatures.

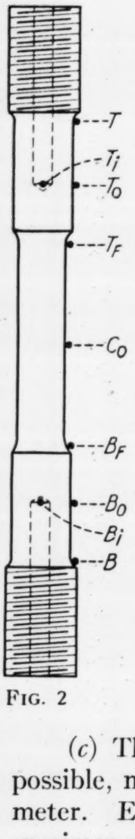

FIG. 2

Location of Thermocouples

T At end of thread
T_i Inside of hole $\frac{1}{2}''$ beyond fillet
T_o Outside $\frac{1}{2}''$ beyond fillet
T_F At fillet
C_o Outside center of gage length
B_F At fillet
B_o Outside $\frac{1}{2}''$ beyond fillet
B_i Inside of hole $\frac{1}{2}''$ beyond fillet
B At end of thread

	Nominal Test Temperature, deg. Fahr.									
T										
T_i										
T_o										
T_F										
C_o										
B_F										
B_o										
B_i										
B										

FORM I.—Form for Reporting Temperature Survey.

(*c*) The transverse dimensions of the furnace should be as small as possible, merely large enough to allow for the shackles and extensometer. Equalization of the temperature over the length of the test specimen shall be obtained by a metallic lining inside the furnace tube, or by the use of multiple heating coils, or external shunts or other adequate means of compensation.

NOTE.—Liquid baths may be used at lower temperatures provided that no reaction of the liquid upon the test metal occurs. Oil is suitable at lower temperatures. No definite recommendations are made as to the length of the furnace, provided the required uniformity of temperature is realized. Reference to the various publications of the Joint Research Committee is suggested for guidance.

Temperature Measuring Apparatus

6. Temperature measurements shall be made by means of suitable thermocouples and potentiometer or millivoltmeter equipment, which shall be accurate to within the following permissible variations for the temperature ranges shown:

	PERMISSIBLE VARIATIONS
Up to and including 1200 F.	± 3 F.
Over 1200 to and including 1600 F.	± 5 F.
Over 1600 F.	± 10 F.

Temperature Survey of Furnace

7. (*a*) A survey of the temperature distribution throughout the test specimens shall be made before making a series of tests of specimens of a given metal, on each furnace and test shackle arrangement used. This survey shall be made using the coupons that are to be tested. A survey of temperature distribution shall be made for each temperature at which specimens shall be tested, and detailed results of these surveys are to be reported with the results of specimen tests. These surveys furnish data for the study of specimen and furnace temperatures. The temperature during the survey shall be also determined at all points indicated in Fig. 2.

(*b*) During a survey of temperature distribution, all temperature readings shall be taken as nearly simultaneously as possible.

Temperature Control During Tension Test

8. (*a*) The temperature of the specimen during the actual tension test shall be measured and recorded. In all cases, a thermocouple shall be located at point C_o, and it is preferred that thermocouples be also located at point T_o or T_i and at point B_o or B_i; these locations are as shown in Fig. 2. Where external contact is made between thermocouple bead and the specimen, the thermocouple shall be shielded in like manner in the tension tests and in the calibration.

(*b*) The change in temperature over the gage length throughout the tension test should be limited to ± 10 F. from the nominal test temperature.

Room Temperature During Test

9. During the period of time required for survey tests and tests of specimens of a metal the range of room temperature shall not exceed 10 F. During all tests the equipment shall be shielded from abnormal drafts.

Temperature Equilibrium

10. Both in survey tests and tests of specimens to fracture great care shall be taken, after heating to the desired temperature, to be

assured that thermal equilibrium has been reached before temperatures are recorded or load and strain readings are taken. In no case shall this heating period be less than 1 hr. The current used in heating the specimen up to the desired temperature shall be approximately that required to hold it at that temperature.

Strain Measurement During Test

11. There shall be reported with the results of all tests, the construction details of the extensometer used, the method of attaching it, and any optical methods for taking strain measurements. The extensometer shall read directly to 0.0002 in. or less for a 2-in. specimen. Whenever feasible, the extensometer should be attached to the gage length of the test specimen, but it is permissible to attach the extensometer to the ends of the shackles projecting beyond the furnace. It is also permissible to employ suitable optical methods of measurement.

NOTE.—Extensometers attached to the rods or shackles beyond the furnace apparently give satisfactory results as to yield strength; but in the determination of elastic modulus, the extension should be taken over a definite portion of the gage length.

Test Procedure

12. (*a*) After thermal equilibrium has been established at the temperature desired for the test of a specimen, a series of load and extensometer readings shall be taken under increasing load until the yield strength has been passed.

(*b*) During the part of the test in which the extensometer is in place the speed of head of the testing machine shall in no case exceed 0.1 in. per minute, and, during the period when readings of load and stretch are being taken, the head of the testing machine shall be stopped or its speed so reduced that simultaneous load and extensometer readings may be taken accurately. Within the range of stretch up to the yield strength, at least ten increments of load (or stretch) shall be taken, and as many more as may be necessary to obtain sufficient data for plotting the points for a good stress-strain diagram. If possible, the speed of head should be so regulated as to require about 10 seconds to apply an increment of load (or stretch). If it is found necessary to stop the motion of the head of the testing machine while taking load and extensometer readings, such readings shall be taken as rapidly as is consistent with accuracy. The time consumed in taking the readings shall be reported with the results.

(*c*) After the yield strength has been passed, the extensometer shall be removed and the test continued to rupture, noting the maximum load carried by the specimen. The speed of the head of the testing machine during this period shall be at a rate of not less than 0.22 nor more than 0.28 in. per minute. After the specimen has ruptured, the elongation in the 2-in. gage length and the reduced diameter at rupture shall be measured as described in Sections 29 and 30 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials.[1] The location and description of the fracture shall be noted.

Calculation of Stress and Strain

13. For any given test load the stress shall be determined by dividing the maximum load by the area of original cross-section of the test specimen. The strain shall be determined by dividing the elongation in the gage length by the original gage length of the specimen.

Recording and Reporting Data

14. A uniform system of recording and reporting of data shall be used. The accompanying forms for recording and reporting shall be used so that results from cooperating laboratories may be readily compared.

(*a*) *Reporting Temperature Surveys.*—For reporting the results of the temperature distribution surveys, Form I is recommended.

(*b*) *Stress-Strain Diagrams.*—Stress-strain diagrams of all tests of specimens shall be included in the report. For plotting stress-strain diagrams[2] of tests the following scales are recommended:

YIELD STRENGTH VALUES, LB. PER SQ. IN.	SCALE FOR STRESS (ORDINATES)	SCALE FOR STRAIN (ABSCISSAS)
70 000 to 140 000	1 in. = 20 000 lb. per sq. in.	1 in. = 0.002 in. per in.
35 000 to 70 000	1 in. = 10 000 lb. per sq. in.	1 in. = 0.001 in. per in.
Below 35 000	1 in. = 5 000 lb. per sq. in.	1 in. = 0.0005 in. per in.

The *yield strength* for each specimen tested shall be determined from its stress-strain diagram. Yield strength is defined as the stress at which a material exhibits a specified limiting permanent set. For short-time high-temperature tension tests of iron, steel and the stronger non-ferrous metals the specified limiting permanent set shall be taken as 0.2 per cent of the gage length of the specimen, and the "offset method" of determining yield strength, as described in Section 26 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials[1] shall be used.

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[1] 1936 Book of A.S.T.M. Standards, Part I, p. 833.

[2] Dietzgen No. 340–10 or equivalent cross-section paper is recommended for this purpose.

(*c*) *Reporting Test Data.*—For reporting the data and results of short-time high-temperature tension tests Form II is recommended.

(*d*) *Plotting Test Data.*—For showing the general results of a series of short-time high-temperature tension tests a set of graphs[1] is recommended, in which temperature values are plotted as abscissas and values of (*1*) tensile strength, (*2*) yield strength, (*3*) elongation in 2 in., and (*4*) reduction of area are plotted as ordinates. For steel and the stronger non-ferrous metals the following scales for plotting are recommended:

FORM II.—FORM FOR REPORTING DATA AND RESULTS OF SHORT-TIME HIGH-TEMPERATURE TENSION TESTS.[a]

Material.. From..

Testing Machine.................................... Extensometer..

Date.. Test Laboratory.....................................

1. RESULTS OF TEMPERATURE DISTRIBUTION SURVEY

Specimen................................						
Nominal test temperature, deg. Fahr........						
Inside temperature, end of specimen beyond fillet, deg. Fahr........................						
Inside temperature center of length of specimen, deg. Fahr.						

2. OBSERVED DATA OF SHORT-TIME HIGH-TEMPERATURE TENSION TESTS

Speed of head of testing machine up to removal of extensometer, in. per minute.....						
Speed of head of testing machine after removal of extensometer, in. per minute.....						
Original diameter of specimen, in............						
Reduced diameter of specimen, in...........						
Maximum load, lb........................						
Elongation in 2 in., in......................						
Type of fracture..........................						
Location of fracture.......................						

3. CALCULATED RESULTS OF SHORT-TIME HIGH-TEMPERATURE TENSION TESTS

Tensile strength, lb. per sq. in.............						
Yield strength (0.2 per cent of set), lb. per sq. in..................................						
Elongation in 2 in., per cent................						
Reduction of area, per cent................						

[a] The above form on letter size sheets suitable for recording test data is available in pads of 25 sheets at 50 cents per pad; 3 pads, $1.00. Address, American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

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[1] Dietzgen No. 340–10 or equivalent cross-section paper is recommended for this purpose.

		TENSION TEST SCALE (ORDINATES)	TEMPERATURE SCAL (ABSCISSAS)
Tensile strength and Yield strength	Max. tensile strength not over 100,000 lb. per sq. in.	1 in. = 10,000 lb. per sq. in.	1 in. = 200 F.
	Max. tensile strength over 100,000 lb. per sq. in.	1 in. = 20,000 lb. per sq. in.	1 in. = 200 F.
Elongation and Reduction of area		1 in. = 10 per cent	1 in. = 200 F.

(*e*) *Summary of Data.*—In reporting results of short-time high-temperature tension tests full information about material, apparatus and test methods shall be reported, as follows:

Materials.—The report shall include the following information concerning the materials tested:

Process of manufacture and whether material is rolled, forged or cast.

Complete chemical analysis.

Complete details of heat treatment.

Physical properties of the material at room temperature.

Size of sample from which specimens were cut.

Relative location and direction of specimen. (Illustrate with sketch.)

If material was removed from service, complete details of service conditions shall be given.

Microstructure and deep etch of samples.

Test Methods.—The report shall include the following information concerning the apparatus and methods of testing:

Description of apparatus, shackles used, method of calibrating loading mechanism and accuracy of loading.

Description of temperature control-recording arrangement.

Description of furnace.

Description of elongation measuring device.

Radical departures, if any, from recommended procedure and technique.

Results of temperature surveys of furnace, Form I (see Paragraph (*a*)).

Stress-strain diagrams (see Paragraph (*b*)).

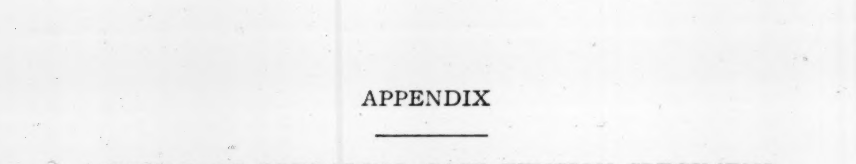
Test data required on Form II (see Paragraph (*c*)).

Curves showing relation of physical properties to temperatures (see Paragraph (*d*)).

APPENDIX

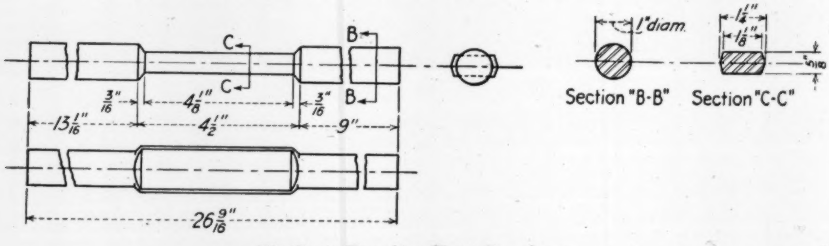
METHOD OF PREPARING CAST TENSION SPECIMENS OF HEAT-RESISTANT METALS

A1. *Scope.*—This appendix covers a method of preparation of cast tension specimens of heat-resistant metals (chromium-nickel, chromium-nickel-iron, and chromium irons) intended for short-time tension tests up to 2200 F.


1 5/8"
Gate
A
1 1/2" rad
1" rad
1" rad
2 1/2" rad
2" rad
1/16" rad
7/8"
5/8"
1/4"
1/8"
7/8"
11/16"
1"
1 1/2"
1 1/2"
1"
5"
Section "A-A"
2 5/16"
7 3/4"
1"
12 3/8"
1"
4"
29 1/4"
1 7/8" diam.
Sprew
1 1/2" diam.
Parting Line
Pattern on Board 3/4" Thick
4 1/8"
11/16"
1 5/8"
4"
4"
Riser
2 9/16"
1/2"
3 15/16"
3/16"
4 1/8"
4 1/2"
5/16"
3/16"
13 1/16"
9"
26 9/16"
1" diam.
1 7/8"
1/2"
13/16"
1 1/2" rad
4 1/4"
4 13/16"
1/8" rad
2 1/2"
2 1/2"
5"

Note-Flask may be Tilted and the Locations, Shape, & Dimensions of Risers may be Modified if Necessary to Improve Quality of Test

(*a*) Details of Pattern Arrangement for Casting Tension Specimen.


C
C
B
B
3/16"
4 1/8"
3/16"
13 1/16"
4 1/2"
9"
26 9/16"
1" diam.
1 1/4"
1/8"
5/16"
Section "B-B"
Section "C-C"

(*b*) Cast Tension Test Specimen.

FIG. A1.—Details of Casting Tension Test Specimen for Heat-Resistant Metals.

A2. *Casting Pattern.*—Structural details of the tension specimen and pattern are given in Fig. A1. The total length of the specimen, Fig. A1 (*a*), is $26\frac{9}{16}$ in. and it is 1 in. round. The center $4\frac{1}{2}$-in. section is rectangular in shape, being $1\frac{1}{4}$ in. in width and $\frac{5}{8}$ in. in thickness.

The gates and risers are so placed with respect to this section as to insure a high degree of soundness. The particular design shown under Fig. A1 (*b*) has proven satisfactory for 35–15 nickel-chromium-iron alloys.[1] Changes in the gates and risers may be advisable for other types of alloys. The soundness of the resulting castings shall be verified by radiographic examination.

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[1] Tests by three foundries have shown that sound castings were produced consistently through the use of this pattern. Tension tests conducted by three laboratories at 1600 F. on these castings showed that the physical properties were in satisfactory agreement.

A3. *Casting Specimen.*—The gage section of the test specimen shall be machined from the central rectangular portion of the casting. Its dimensions shall conform to those given in Fig. 1 of this method. The shanks of the cast specimens are purposely made of sufficient length to remove all threaded or other connections from the heated zones of the test furnace. This procedure is necessary since specimens of this type will in general be tested at temperatures up to 2200 F. The ends of the shanks may be threaded to fit the adapters of the testing machine. The specimen shall be machined true and axial.

A4. *Testing Apparatus and Procedure.*—All details as to the tension machine and test procedure shall be in accordance with those specified in this method.

TENTATIVE DEFINITIONS OF TERMS RELATING TO RHEOLOGICAL PROPERTIES OF MATTER[1]

A.S.T.M. Designation: E 24 - 37 T

This is a **Tentative Standard** and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

ISSUED, 1934; REVISED 1937.[2]

Consistency.—The resistance to deformation of material. Quantitatively it is the ratio of the shearing stress to the rate of shear.

Plasticity.—The ability of a body to retain a part or all of its deformation after reduction of the deforming stress to zero.

Elasticity.—That property of a body by virtue of which it recovers its original size and shape after deformation.

Liquids.—Substances which undergo continuous deformation when subjected to shearing stress.

Simple Liquids.—Those substances in which the rate of shear is proportional to the shearing stress. The constant ratio of shearing stress to rate of shear of a simple liquid is the *viscosity* of the liquid.

Complex Liquids.—Those substances in which the rate of shear is not proportional to the shearing stress.

Solids.—Substances which undergo permanent deformation only when subjected to shearing stress in excess of some finite value characteristic of the substance.

Plastic Solids.—Those substances which do not deform under a shearing stress until the stress attains a critical value (yield stress) when the solids deform so readily or so quickly that, except for inertia effects, the stress never exceeds the yield stress.

Elastic Solids.—Those substances in which, for all values of the shearing stress below the rupture stress (shear strength), the strain is fully determined by the stress regardless of whether the stress is increasing or decreasing.

NOTE.—Most solids exhibit combinations of properties. Metals, for example, are mainly elastic-plastic, while some petroleum residues, waxes and similar materials are plastic-viscous. Some materials exhibit even more complex deformation, for example, rubber compounds.

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[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

[2] Revision accepted by Committee E-10 on Standards, October 11, 1937.

TENTATIVE REVISIONS
OF
A.S.T.M. STANDARDS

Tentative revisions of A.S.T.M. Standards are printed for one or more years with a view of eliciting criticisms, of which the committee concerned will take due cognizance before recommending final action.

Members of the Society and others are invited to direct written criticisms of any of these Tentative Revisions to the Headquarters of the Society, 260 So. Broad St., Philadelphia, Pa.

TENTATIVE REVISIONS OF A.S.T.M. STANDARDS

Tentative Revision[1] of Standard Specifications for Boiler and Firebox Steel for Locomotives (A.S.T.M. Designation: A 30 – 33):[2]

Section 6.—Change the table of physical properties appearing in this section from its present form: namely,

	Flange	Firebox
Tensile strength, lb. per sq. in...	55 000 to 65 000	52 000 to 62 000
Yield point, min., lb. per sq. in..	0.5 tens. str.	0.5 tens. str.
Elongation in 8 in., min., per cent	$\frac{1\,500\,000}{\text{tens. str.}}$[a]	$\frac{1\,550\,000}{\text{tens. str.}}$[a]

[a] See Section 7.

to read as follows:

	Flange	Firebox	
		Grade A	Grade B
Tensile strength, lb. per sq. in.	55 000 to 65 000	52 000 to 62 000	48 000 to 58 000
Yield point, min., lb. per sq. in.	0.5 tens. str.	0.5 tens. str.	0.5 tens. str.
Elongation in 8 in., min., per cent....................	$\frac{1\,500\,000}{\text{tens. str.}}$[a]	$\frac{1\,550\,000}{\text{tens. str.}}$[a]	$\frac{1\,550\,000}{\text{tens. str.}}$[a]

[a] See Section 7.

Revision submitted, August, 1937.

Tentative Revision[1] of Standard Specifications for High Tensile Strength Carbon-Steel Plates for Pressure Vessels (Plates 2 in. and Under in Thickness) (A.S.T.M. Designation: A 149 – 36):[2]

Title.—Change the title to read as follows by the addition of the italicized words:

Standard Specifications for High Tensile Strength Carbon-Steel Plates for *Boilers and Other* Pressure Vessels (Plates 2 in. and Under in Thickness).

Revision submitted, June, 1937.

Section 1.—Change this section from its present form: namely,

1. These specifications cover two grades of high tensile strength carbon-steel plates up to 2 in., inclusive, in thickness, of flange and firebox qualities, for boilers and other pressure vessels. This material is suitable for fusion welding. The term grade is used to distinguish between different tensile ranges.

to read as follows:

1. These specifications cover two high tensile strength ranges of carbon-steel plates, designated Grades A and B; up to 2 in., inclusive, in thickness; of flange and ordinary firebox qualities; for use in locomotive boiler shells, boilers for stationary service and other pressure vessels. This material is suitable for fusion welding. A definite silicon content is specified in order to limit the carbon to the lowest practicable amount consistent with the specified tensile strength and the thickness of the material. Welding technique is of fundamental importance and it is presupposed that welding procedure will be in accordance with approved methods.

Revision submitted, June, 1937.

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part I.

Section 3.—Change this section from its present form: namely,

3. The steel shall conform to the following maximum requirements as to chemical composition:

		Grade A	Grade B
Carbon, per cent	for plates ¾ in. and under in thickness	0.30	0.32
	for plates over ¾ in. in thickness	0.33	0.35
Manganese, per cent		0.90	0.90
Phosphorus, per cent	Flange	0.04	0.04
	Firebox	0.035	0.035
Sulfur, per cent	Flange	0.05	0.05
	Firebox	0.04	0.04
Silicon, per cent		0.25	0.25

to read as follows:

3. The steel shall conform to the following requirements as to chemical composition:

		Grade A	Grade B
Carbon, max., per cent	for plates 1 in. and under in thickness	0.28	0.31
	for plates over 1 to 2 in., incl., in thickness	0.31	0.33
Manganese, max., per cent		0.90	0.90
Phosphorus, max., per cent	Flange	0.04	0.04
	Firebox	0.035	0.035
Sulfur, max., per cent	Flange	0.05	0.05
	Firebox	0.04	0.04
Silicon, per cent		0.15 to 0.30	0.15 to 0.30

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for High Tensile Strength Carbon-Steel Plates for Fusion-Welded Pressure Vessels (Plates over 2 in. to 4 in., Inclusive, in Thickness) (A.S.T.M. Designation: A 150 – 36):[2]

Title.—Change the title to read as follows by the addition of the italicized words and figure and the omission of the words in brackets:

Standard Specifications for High Tensile Strength Carbon-Steel Plates for [Fusion-Welded] *Boilers and Other* Pressure Vessels (Plates over 2 [in.] to 4 *1/2* in., Inclusive, in Thickness).

Revision submitted, June, 1937.

Section 1.—Change this section from its present form: namely,

1. These specifications cover two grades of high tensile strength carbon-steel plates over 2 in. to 4 in., inclusive, in thickness; of firebox quality; for fusion-welded boilers and other pressure vessels. The term grade is used to distinguish between different tensile ranges.

to read as follows:

1. These specifications cover two high tensile strength ranges of carbon-steel plates, designated as Grades A and B, over 2 to 4½ in., inclusive, in thickness but not exceeding 25,000 lb. theoretical weight for plates over 4¼ to 4½ in., inclusive, in thickness; of ordinary firebox quality; for use in boilers and other pressure vessels. This material is suitable for fusion welding. A definite silicon content is specified in order to limit the carbon to the lowest practical amount consistent with the specified tensile strength and the thickness of the material. Welding technique is of fundamental

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part I.

importance and it is presupposed that welding procedure will be in accordance with approved methods.

Revision submitted, June, 1937.

Section 3 (b).—Change the last sentence of this section to read as follows by the addition of the italicized words and the omission of those in brackets:

Pieces for two [complete sets of such tests] ***tension tests (one top and one bottom)*** shall be provided from each plate as rolled.

Revision submitted, June, 1937.

Section 4.—Change this section from its present form: namely,

4. The steel shall conform to the following requirements as to chemical composition:

Carbon, max., per cent		0.35
Manganese, per cent		0.50 to 0.90
Phosphorus, max., per cent	Acid	0.04
	Basic	0.035
Sulfur, max., per cent		0.04
Silicon, max., per cent		0.25

to read as follows:

4. The steel shall conform to the following requirements as to chemical composition:

	GRADE A	GRADE B
Carbon, max., per cent	0.33	0.35
Manganese, per cent	0.50 to 0.90	0.50 to 0.90
Phosphorus (Basic), max., per cent	0.035	0.035
Sulfur, max., per cent	0.04	0.04
Silicon, per cent	0.15 to 0.30	0.15 to 0.30

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for Structural Rivet Steel (A.S.T.M. Designation: A 141 – 36):[2]

Section 9.—Change this section from its present form: namely,

9. The diameter of rivet bars shall not vary from the size ordered more than the amounts given in the following table:

	PERMISSIBLE VARIATIONS, IN.	
DIAMETER	OVER	UNDER
Up to and including $\frac{1}{2}$ in	0.007	0.007
Over $\frac{1}{2}$ up to and including 1 in	0.010	0.010
Over 1 up to and including 2 in	$\frac{1}{32}$	$\frac{1}{64}$

to read as follows:

9. The diameter of rivet bars shall not vary from the size ordered by more than the amounts given in the following table:

	PERMISSIBLE VARIATIONS IN DIAMETER, IN.		
DIAMETER SPECIFIED, IN.	UNDER	OVER	OUT OF ROUND
Up to $\frac{5}{16}$, incl	0.007	0.007	0.010
Over $\frac{5}{16}$ to $\frac{7}{16}$, incl	0.007	0.007	0.011
Over $\frac{7}{16}$ to $\frac{5}{8}$, incl	0.008	0.008	0.012
Over $\frac{5}{8}$ to $\frac{7}{8}$, incl	0.009	0.009	0.014
Over $\frac{7}{8}$ to 1, incl	0.010	0.010	0.015
Over 1 to $1\frac{1}{8}$, incl	0.012	0.012	0.016
Over $1\frac{1}{8}$ to $1\frac{1}{4}$, incl	0.014	0.014	0.018
Over $1\frac{1}{4}$ to $1\frac{3}{8}$, incl	0.016	0.016	0.022
Over $1\frac{3}{8}$ to $1\frac{1}{2}$, incl	0.018	0.018	0.026
Over $1\frac{1}{2}$ to 2, incl	0.022	0.022	0.030

Revision submitted, June, 1937.

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part I.

Tentative Revision[1] of Standard Specifications for Alloy-Steel Bolting Material for High-Temperature Service (A.S.T.M. Designation: A 96 – 33):[2]

Explanatory Note.—Change the Explanatory Note, appearing at the end of these specifications, to read as follows:

NOTE.—Allowable pressures and their corresponding allowable temperatures are given in the following table. Higher temperatures with appropriate pressures may be used at the discretion of the designing engineer.

TABLE I.—MAXIMUM SERVICE PRESSURES FOR POWER PIPING AND OIL PIPING AT TEMPERATURES BELOW AND ABOVE 750 F.

Primary Service Pressure Rating at 750 F., lb. per sq. in.	100	300	400	600	900	1500
SERVICE TEMPERATURE, DEG. FAHR.	MAXIMUM SERVICE PRESSURES, LB. PER SQ. IN.					
POWER PIPING AND OIL PIPING						
100	230	500	670	1000	1500	2500
150	220	480	640	960	1440	2400
200	210	465	620	930	1395	2325
250	200	450	600	900	1350	2250
300	190	435	580	870	1305	2175
350	180	420	560	840	1260	2100
400	170	405	540	810	1215	2025
450	160	390	520	780	1170	1950
500	150	375	500	750	1125	1875
550	140	360	480	720	1080	1800
600	130	345	460	690	1035	1725
650	120	330	440	660	990	1650
700	110	315	420	630	945	1575
750	100	300	400	600	900	1500
POWER PIPING						
800	85	250	335	500	750	1250
850	70	200	270	400	600	1000
OIL PIPING						
800	92	275	370	550	830	1380
850	82	245	330	490	740	1230
900	70	210	280	420	630	1050
950	55	165	220	330	495	825
1000	40	120	160	240	360	600

Reference may be made to the dimensional standards for hydraulic pressure ratings and corresponding temperatures formulated by the Sectional Committee on Standardization of Pipe Flanges and Fittings, under the procedure of the American Standards Association, A.S.A. Project B 16 and more specifically to the American Standard for Steel Flanged Fittings and Companion Flanges, B 16*e* – 1932. Copies of this American Standard are obtainable from the American Standards Association or from the American Society of Mechanical Engineers, 29 West 39th St., New York City.

Revision submitted, June, 1934; Modified, August, 1934.

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part I.

Section 16.—Change this section to read as follows by the addition of the italicized words, the chemical requirements remaining the same:

16. Bolts and studs shall be equipped with semifinished nuts of American Standard heavy dimensions, chamfered and trimmed. *Nuts shall be made by the cold-punched or hot-forged, cold-trimmed process; nuts cut from drawn or rolled bar stock shall not be permitted.* If washers are used under nuts, they shall be of forged or rolled steel. All nuts and washers shall be free from injurious defects and shall have a workmanlike finish. Nuts shall conform to the chemical requirements for phosphorus and sulfur as follows.

Revision submitted, June, 1934.

Section 17.—Change this section to read as follows by the addition of the italicized words:

17. American National Standard coarse screw threads shall be used, except that no stud *or bolt* shall have less than eight threads per inch.

Revision submitted, June, 1934.

Tentative Revision[1] of Standard Specifications for Iron and Steel Chain (A.S.T.M. Designation: A 56 – 30):[2]

Section 1.—Change the second paragraph to read as follows by the addition of the italicized word and the omission of the word in brackets:

Crane Chain, for slings, [hoists] *cranes*, steam shovels, and marine uses, and where an all-iron chain is desired.

Revision submitted, August, 1937.

Section 2.—Change the paragraph covering crane chain to read as follows by the addition of the italicized words:

Crane Chain.—Crane Chain shall be made from *all pig puddled or processed* wrought iron free from any admixture of iron scrap or steel.

Revision submitted, June, 1937.

Change the paragraph covering proof coil from its present form: namely,

Proof Coil.—Proof coil may be made from open-hearth steel or all reworked scrap.

NOTE.—The term "reworked scrap" applies to steel and iron scrap, or a mixture of both.

to read as follows, omitting the note defining "reworked scrap:"

Steel Proof Coil.—Proof coil shall be made from open-hearth steel.

Revision submitted, August, 1937.

Section 4.—Change the table of requirements as to chemical composition to read as follows by the addition of the italicized figure and the omission of the figure in brackets:

	WROUGHT IRON CRANE CHAIN	STEEL PROOF COIL
Manganese, maximum, per cent	[0.10] *0.06*	....
Phosphorus, maximum, per cent	....	0.05
Sulfur, maximum, per cent	....	0.05

Revision submitted, August, 1937.

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part I.

Table I.—Change the present proof test loads for crane chain wrought iron appearing in the second column of Table I by substituting the values shown in the following table under Proposed Revised Proof Test Loads; the new requirements being 38 per cent of the breaking load:

NOMINAL SIZE OF CHAIN BAR, IN.	CRANE CHAIN WROUGHT IRON PRESENT PROOF TEST LOADS	 PROPOSED REVISED PROOF TEST LOADS
$\frac{1}{4}$	1 767	1 345
$\frac{5}{16}$	2 760	2 100
$\frac{3}{8}$	3 975	3 020
$\frac{7}{16}$	5 415	4 115
$\frac{1}{2}$	7 072	5 375
$\frac{9}{16}$	8 947	6 800
$\frac{5}{8}$	11 047	8 395
$\frac{3}{4}$	15 900	12 080
$\frac{7}{8}$	21 622	16 430
1	28 275	21 490
$1\frac{1}{8}$	33 400	25 380
$1\frac{1}{4}$	41 250	31 350
$1\frac{3}{8}$	49 900	37 920
$1\frac{1}{2}$	59 350	45 100
$1\frac{5}{8}$	69 750	53 000
$1\frac{3}{4}$	80 800	61 400
$1\frac{7}{8}$	92 750	70 500
2	105 500	80 200

Revision submitted, August, 1937.

New Section.—Add the following requirements for marking as a new Section 14 renumbering the present Sections 14 to 16 accordingly:

14. *Marking.*—No markings shall be stamped on any links of the chain. Metal tags with the words, "Crane" or "Proof Coil" stamped thereon shall be securely attached to each end of each chain.

Revision submitted, August, 1937.

Tentative Revision[1] of Standard Specifications for Welded Wrought-Iron Pipe (A.S.T.M. Designation: A 72 – 33):[2]

Section 2.—Change Paragraph (*a*) to read as follows by the addition of the italicized words:

2. (*a*) The pipe shall be made from *all pig puddled or processed* wrought iron and shall be free from any admixture of iron scrap or steel.

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for Wrought-Iron Rolled or Forged Blooms and Forgings (A.S.T.M. Designation: A 73 – 36):[2]

Section 2.—Change the first sentence of Paragraph (*a*) to read as follows by the addition of the italicized words:

Blooms and forgings of Class A shall be made from *all pig puddled or processed* wrought iron and shall be free from any admixture of iron scrap or steel.

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for Staybolt Wrought Iron, Solid (A.S.T.M. Designation: A 84 – 36):[2]

Section 2.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part I.

2. The bars shall be rolled from a [bloom,] slab pile or box pile made from [reworked wrought iron] ***all pig puddled***, [or reworked] knobbled charcoal ***or processed wrought*** iron. The original muck [or knobbled] bars shall be [twice] piled ***rolled, repiled*** and rerolled. The [wrought iron and] component parts of the [bloom,] slab pile or box pile shall be free from any admixture of iron scrap or steel.

Revision submitted, June, 1937.

Section 3.—Omit Paragraph (*a*), which reads as follows, relettering the present Paragraphs (*b*) and (*c*) as Paragraphs (*a*) and (*b*), respectively:

3. (*a*) ***Bloom.***—A bloom is a solid mass of iron that has been hammered into a convenient size for rolling.

Revision submitted, June, 1937.

Section 11.—Add a new Paragraph (*b*) to read as follows, relettering the present Paragraph (*b*) as Paragraph (*c*):

(*b*) Where small lots of different sizes are to be tested, the bas shall be grouped into lots of 50, or fraction thereof, without regard to size, and one bar representing the lot shall be tested as specified in Sections 6, 8 and 9.

Revision submitted, June, 1937.

Section 12.—Change this section from its present form: namely,

12. The bars shall be truly round within 0.01 in. The diameter shall be not less than 0.005 in. over nor more than 0.020 in. over the specified diameter.

to read as follows:

12. (*a*) All bars shall be truly round within 0.010 in., and shall conform to the following requirements as to permissible variations in size:

NOMINAL DIAMETER OF BAR, IN.	MINIMUM	MAXIMUM	TOTAL ROLLING TOLERANCE
BARS FOR THREADING			
7/8	0.895	0.910	0.015
15/16	0.958	0.973	0.015
1	1.020	1.035	0.015
1 1/16	1.083	1.098	0.015
1 1/8	1.145	1.160	0.015
1 3/16	1.208	1.223	0.015
1 1/4	1.270	1.285	0.015
BARS FOR FORGING			
7/8	0.880	0.895	0.015
15/16	0.943	0.958	0.015
1	1.005	1.020	0.015
1 1/16	1.068	1.083	0.015
1 1/8	1.130	1.145	0.015
1 3/16	1.193	1.208	0.015
1 1/4	1.255	1.270	0.015
1 5/16	1.318	1.333	0.015
1 3/8	1.380	1.395	0.015
1 7/16	1.443	1.458	0.015
1 1/2	1.505	1.520	0.015
1 5/8	1.630	1.645	0.015
1 3/4	1.755	1.770	0.015
1 7/8	1.880	1.895	0.015
2	2.005	2.020	0.015

Revision submitted, August, 1937.

Tentative Revision[1] of Standard Specifications for Staybolt Wrought Iron, Hollow Rolled (A.S.T.M. Designation: A 86 – 36):[2]

Make the same changes in Sections 2, 3 and 11 of these specifications as are recommended in Specifications A 84, above.

Revision submitted, June, 1937.

Make the same change in Section 12 (*a*) of these specifications as is recommended in Specifications A 84, above.

Revision submitted, August, 1937.

Tentative Revision[1] of Standard Specifications for Uncoated Wrought-Iron Sheets (A.S.T.M. Designation: A 162 – 36):[2]

Section 2.—Change to read as follows by the addition of the italicized words:

2. All sheets shall be rolled from slabs, piles or bars made from *all pig puddled, knobbled charcoal or processed* wrought iron and shall be free from any admixture of iron scrap (Note) or steel.

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for Zinc-Coated (Galvanized) Wrought-Iron Sheets (A.S.T.M. Designation: A 163 – 36):[2]

Section 2.—Change to read as follows by the addition of the italicized words:

2. All sheets shall be rolled from slabs, piles or bars made from *all pig puddled or processed* wrought iron and shall be free from any admixture of iron scrap (Note) or steel.

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (A.S.T.M. Designation: A 116 – 30):[2]

The Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (A 116 – 37 T)[3] are in effect a tentative revision of and are intended to replace when adopted the present Standard Specifications A 116 – 30.

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (A.S.T.M. Designation: A 121 – 30):[2]

The Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (A 121 – 37 T)[4] are in effect a tentative revision of and are intended to replace when adopted the present Standard Specifications A 121 – 30.

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for Manganese-Bronze Ingots for Sand Castings (A.S.T.M. Designation: B 7 – 27):[2]

Section 3.—Change the table of chemical requirements appearing in this section to read as follows by the addition of the italicized words and figures and the omission of the figures in brackets:

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part I.

[3] See p. 646.

[4] See p. 643.

	MAXIMUM	MINIMUM
Copper, per cent	[60] *62.0*	55.0
Zinc, per cent	[42] *remainder*	[38] *remainder*
Tin, per cent	1.5	0.0
Manganese, per cent	3.5	[0.0] *0.2*
Aluminum, per cent	1.5	[0.0] *0.5*
Iron, per cent	2.0	[0.0] *0.6*
Lead, per cent	[0.2] *0.4*[a]	0.0

[a] In the case of the Specifications for Castings (B 54) the present requirement for lead is 0.4 per cent; the proposed change is intended to apply only to the lead content requirement in the Specifications for Ingots (B 7).

Revision submitted, June, 1936.

Tentative Revision[1] of Standard Specifications for Manganese-Bronze Sand Castings (A.S.T.M. Designation: B 54 – 27):[2]

Section 3.—Make the same change in this section as recommended in Section 3 of Specifications B 7 – 27, above.

Revision submitted, June, 1936.

Tentative Revision[1] of Standard Specifications for Rolled Zinc (A.S.T.M. Designation: B 69 – 29):[2]

The Tentative Specifications for Rolled Zinc (B 69 – 37 T)[3] are in effect a tentative revision of and are intended to replace when adopted the present Standard Specifications B 69 – 29.

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for Quicklime for Structural Purposes (A.S.T.M. Designation: C 5 – 26):[4]

The Tentative Specifications for Quicklime for Structural Purposes (C 5 – 34 T)[5] are in effect a tentative revision of and are intended to replace when adopted the present Standard C 5 – 26.

Revision submitted, June, 1934.

Tentative Revision[1] of Standard Specifications for Hydrated Lime for Structural Purposes (A.S.T.M. Designation: C 6 – 31):[4]

The Tentative Specifications for Hydrated Lime for Structural Purposes (C 6 – 34 T)[6] are in effect a tentative revision of and are intended to replace when adopted the present Standard C 6 – 31.

Revision submitted, June, 1934.

Tentative Revision[1] of Standard Specifications for Concrete Sewer Pipe (A.S.T.M. Designation: C 14 – 35):[2]

Section 1.—Add the following footnote:

Caution.—The consumer or purchaser is cautioned against using cement-concrete pipe where the sewage shows an acid reaction.

Revision submitted, June, 1924.

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part I.

[3] See p. 698.

[4] 1936 Book of A.S.T.M. Standards, Part II.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 749 (1934); also 1937 Book of A.S.T.M. Tentative Standards, p. 477.

[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 752 (1934); also 1937 Book of A.S.T.M. Tentative Standards, p. 480.

Tentative Revision[1] of Standard Specifications for Building Brick (Made from Clay or Shale) (A.S.T.M. Designation: C 62 - 30):[2]

The Tentative Specifications for Building Brick (Made from Clay or Shale) (C 62 - 37 T)[3] are in effect a tentative revision of and are intended to replace when adopted the present Standard Specifications C 62 - 30.

Revision submitted, June, 1935; Modified, June, 1936 and June, 1937.

Tentative Revision[1] of Standard Specifications for Curing Portland-Cement Concrete Slabs with Calcium-Chloride Admixture (A.S.T.M. Designation: C 82 - 34):[2]

Title.—Change to read as follows by the omission of the word in brackets:

> Standard Specifications for Curing Portland-Cement Concrete [Slabs] with Calcium-Chloride Admixture.

Revision submitted, June, 1937.

Section 1.—Change to read as follows by the omission of the words in brackets:

> 1. These specifications cover the method of curing [exposed] portland-cement concrete [slabs] by accelerating the setting and hardening of the concrete by means of calcium chloride [solution] incorporated with the mixture.

Revision submitted, June, 1937.

Sections 3 and 4.—Replace these two sections which read as follows:

> 3. A concentrated solution shall be prepared by adding 100 lb. of calcium chloride to 13 gal. of water previously placed in the container. The solution shall be thoroughly stirred until it is of uniform concentration and shall have a specific gravity of from 1.31 to 1.36 at 60 F.
>
> 4. From 2 to 3 pt., according to the rate of set of the concrete, of the calcium chloride solution for each bag of cement (or for each 94 lb. of cement) shall be introduced into the drum of the mixer with the mixing water. A positive automatic mechanical method shall be used for introducing the calcium chloride solution into the mixer drum in company with the water.

by the following new Section 3, renumbering the subsequent sections accordingly:

> 3. *Use of Calcium Chloride.*—Calcium chloride may be added to the mix in either the dry or solution form, as follows:
>
> (*a*) When used in the dry form from 1 to 2 lb. of calcium chloride per bag of cement, according to the temperature[a] prevailing at the time of concreting, shall be placed in the skip with the aggregates but not in contact with the cement, just prior to discharging the contents of the skip into the mixer drum.
>
> [a] The following amounts are recommended: At temperatures below 80 F. (27 C.), 2 lb.; 80 to 90 F. (27 to 32 C.), 1.5 lb.; above 90 F. (32 C.), 1 lb. (1 qt. of solution contains 1 lb. of calcium chloride).
>
> (*b*) When used in the solution form the following procedure shall be followed in the preparation and addition of the solution to the concrete mix: Dissolve one bag of calcium chloride (100 lb.) in a quantity of water sufficient to make 25 gal. of solution (100 qt.). The solution shall be thoroughly stirred until it is of uniform concentration. From 1 to 2 qt. of this solution per bag of cement, according to the temperature[a]

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part II.

[3] See p. 733.

prevailing at the time of concreting, shall be introduced into the drum of the mixer with the mixing water. The quantity of mixing water shall be reduced by the amount of calcium chloride solution used. A positive method shall be used for introducing the calcium chloride solution.

Revision submitted, June, 1937.

Section 5.—Change to read as follows by the addition of the italicized words and figure and the omission of the figure in brackets:

[5]. *4.* The concrete shall be finished and edged promptly after placing, as concrete containing calcium chloride hardens ***more*** rapidly ***than plain concrete.***

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for Curing Portland-Cement Concrete Slabs by Surface Application of Calcium Chloride (A.S.T.M. Designation: C 83 – 36):[2]

Section 4.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

4. (*a*) After removal of the burlap, ***the pavement shall be sprinkled and*** [2] ***1.5*** lb. of calcium chloride shall be ***immediately*** applied to each square yard of the pavement surface. The material shall be spread by means of a squeegee or suitable mechanical device, and [further shoveled or broomed if necessary] ***shall be screeded or belted upon dissolution*** to [secure] ***insure*** uniform [distribution] ***coverage of the concrete slab.*** [Spreading shall not be done with shovels or brooms alone.]

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for Ready Mixed Concrete (A.S.T.M. Designation: C 94 – 35):[2]

Section 14.—Change to read as follows by the omission of the words in brackets:

14. [Coarse] Aggregates shall be measured by weight. [Fine aggregates shall be measured by weight or by inundated volume in a device approved by the purchaser.] Batch weights shall be based on dry materials and shall be corrected to take into account the weight of moisture contained in the aggregates.

Revision submitted, June, 1937.

Section 19.—Add to the marginal heading a reference letter "a" to refer to the accompanying footnote shown below under the new Sections 20 and 21.

Change the sixth sentence of this section to read as follows by the addition of the italicized words and the omission of those in brackets:

When the central mixing plant is [depended upon] ***used*** for the complete mixing ***of concrete to be transported in an agitator truck,*** the minimum mixing time for mixers of 1-cu. yd. capacity or less shall be not less than 1 min.; for larger capacities of mixers this mixing time shall be increased at the rate of 15 sec. or more for each cubic yard, or fraction thereof, additional capacity.

Immediately following the sixth sentence add a new sentence to read as follows:

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part II.

When the central mixing plant is used for the complete mixing of concrete to be transported in a non-agitating truck, the minimum mixing time for mixers of 1-cu. yd. capacity or less shall not be less than 1 min.; for larger capacities of mixers this mixing time shall be increased at the rate of 30 sec. or more for each cubic yard, or fraction thereof, additional capacity.

Revision submitted, June, 1937.

New Sections.—Add as new Sections 20 and 21 the seventh and eighth sentences, respectively, of Section 19, modified to read as follows, renumbering the subsequent sections accordingly:

20. *Agitation.*[a]—When mixed concrete is transported in a truck mixer or an agitator, the size of batch shall not exceed the rated capacity of the equipment for agitation as stated by the manufacturer, and as stamped in metal at a prominent place on the equipment. The concrete shall be agitated at the rate specified by the manufacturer as agitating speed.

21. *Shrink-Mixing.*[a]—When a truck mixer, or an agitator provided with adequate mixing blades is used for transportation, the mixing time at the central mixer may be reduced to the minimum required (about 30 sec.) to incorporate the ingredients of the mixture into a mass, and the mixing completed in the truck mixer or agitator. Under these circumstances, all ingredients for a batch shall be in the central mixer and partially mixed before any concrete is discharged to the truck mixer or agitator, and the size of batch shall not exceed the rated capacity of the equipment for shrink mixing as stated by the manufacturer, and as stamped in metal at a prominent place on the equipment. The mixing time in the truck mixer or agitator shall be as specified for truck mixing in Section 22.

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[a] The Tentative Standards and Recommended Practices for Truck Mixers and Agitators of the Revolving Drum Type of the National Ready Mixed Concrete Association place the following limitations on Capacities and Mixing Speeds:

3. *Capacities.*—(*a*) When used for truck mixing the capacity of top-door-loading truck mixers shall be in accordance with the manufacturer's rating but the volume of mixed concrete shall not exceed 50 per cent of the gross volume of the drum.

(*b*) When used for truck mixing the capacity of end-loading truck mixers shall be in accordance with the manufacturer's rating but the volume of mixed concrete shall not exceed 57.5 per cent of the gross volume of the drum.

(*c*) When used for shrink mixing, the capacity of truck mixers shall be in accordance with the manufacturer's rating but the volume of mixed concrete shall not exceed 66.66 per cent of the gross volume of the drum.

(*d*) When used for agitating, the capacity of the truck mixer or of the truck agitator shall be in accordance with the manufacturer's rating but the volume of mixed concrete shall not exceed 80 per cent of the gross volume of the drum.

(*e*) The manufacturer shall attach to each truck mixer and truck agitator a metal plate on which is stated the capacities, in terms of volume of mixed concrete, for the various uses to which the equipment is applicable. When a capacity less than the limit indicated in Section 3 (*d*) is shown on the metal plate it indicates the maximum safe load carrying capacity for which the equipment was designed.

4. *Mixing Speeds.*—(*a*) Mixing speed shall be not less than 4 r.p.m. of the drum nor greater than a speed resulting in a peripheral velocity of the drum of 225 ft. per min. (10 to 15 r.p.m., depending on size of mixer.)

(*b*) Agitating speed shall be not less than 2 nor more than 6 r.p.m. of the drum.

Revision submitted, June, 1937.

Section 20.—Renumber as Section 22 and add to the marginal heading a reference letter "a" to refer to the accompanying footnote given above under the new Sections 20 and 21.

Revision submitted, June, 1937.

Section 22.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

[22] *24.* Concrete shall be delivered to the site of the work, and discharge from the hauling container shall be completed within a period of 1½ hr. (*Note 1*) after the introduction of the mixing water to the cement and aggregates, or the cement (*Note 2*) to

the aggregate [when the fine aggregate contains moisture in excess of 6 per cent by weight and the coarse aggregate contains moisture in excess of 3 per cent by weight].

Revision submitted, June, 1937.

Immediately following this section add two new notes to read as follows:

NOTE 1.—Research has shown that a period of 1½ hr. between the introduction of mixing water and the completion of the discharge of the batch results in no detrimental effects on the concrete; under specially favorable conditions periods up to 2 and 3 hr. may be allowed. Where air temperatures are unusually high, or the ingredients of the concrete mixture are such that an unusually quick time of set or loss of plasticity may occur, it may be necessary to specify shorter periods to insure that no damage be done to the concrete; under these circumstances an investigation, satisfactory to the engineer, with the materials to be used and in the proposed proportions, should be conducted to determine if a shorter period is necessary. It is unlikely that, except under the most unusual circumstances, it will be necessary to substitute a period of less than 1 hr. for the 1½ hr. applicable to normal conditions.

NOTE 2.—The limitation on the time between the introduction of the cement to the aggregates may be waived when, in the judgment of the engineer, the aggregates are sufficiently free from moisture that there will be no harmful effects on the cement.

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Method of Making and Storing Compression Test Specimens of Concrete in the Field (A.S.T.M. Designation: C 31 – 33):[2]

Section 4.—Add a new Section 4 on Storage Box to read as follows, renumbering the subsequent sections accordingly:

4. *Storage Box.*—In order to afford reasonably uniform temperature and moisture conditions during the first 24 hr., for curing the specimens and to protect them from damage, the cylinder molds shall be placed within a storage box (Note) so constructed and kept in such a position on the work that its air temperature when containing concrete specimens shall remain within 60 to 80 F. (16 to 27 C.). Other suitable means which provide similar temperature and moisture conditions may be used.

NOTE.—It is suggested that the storage box mentioned in Section 4 be made of 1-in. dressed tongue-and-groove lumber, well braced with battens to avoid warping. The box should be well painted inside and outside and should be provided with a hinged cover and padlock.

Revision submitted, June, 1937.

Section 7.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and the omission of those in brackets:

7. (*a*) At the end of 24 hr., test specimens shall be removed from the [place of casting] *storage box* and, if desired, [molds] *the mold* may be removed. Those specimens intended for laboratory control tests shall be placed under moist curing conditions at approximately 70 F. and maintained therein until tested.

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Definitions of Terms Relating to Refractories (A.S.T.M. Designation: C 71 – 36):[2]

Several definitions of terms published under the title "Tentative Definitions of Terms Relating to Refractories (C 71 – 37 T)"[3] are

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part II.

[3] See p. 790.

intended to be added when adopted to the present Standard Definitions C 71 - 36.

Revision submitted, June, 1936; Modified, June, 1937.

Tentative Revision[1] of Standard Definitions of Terms Relating to the Gypsum Industry (A.S.T.M. Designation: C 11 - 28):[2]

Several definitions of terms published under the title "Tentative Definitions of Terms Relating to the Gypsum Industry (C 11 - 31 T)"[3] are intended to be added when adopted to the present Standard Definitions C 11 - 28.

Revision submitted, June, 1916; Modified, June, 1931.

Tentative Revision[1] of Standard Definition of the Term Sand (A.S.T.M. Designation: C 58 - 28):[2]

The Tentative Definition of the Term Aggregate (C 58 - 28 T)[4] is intended to be included, when adopted, with the present Standard Definition C 58 - 28.

Revision submitted, June, 1926; Modified, June, 1928.

Tentative Revision[1] of Standard Specifications for Basic Sulfate White Lead (A.S.T.M. Designation: D 82 - 24):[2]

The Tentative Specifications for Basic Sulfate White Lead (D 82 - 37 T)[5] are in effect a tentative revision of and are intended to replace when adopted the present Standard Specifications D 82 - 24.

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for Zinc Oxide (A.S.T.M. Designation: D 79 - 24):[2]

Section 3.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> 3. (*a*) The color, *tinting strength, brightness* and *oil absorption* [color strength], when specified, shall be equal to that of a sample mutually agreed upon by *the* buyer and *the* seller.

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for Zinc Sulfide (A.S.T.M. Designation: D 386 - 36):[2]

Section 2.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> 2. (*a*) Dry Pigment.—The dry pigment shall consist of zinc sulfide, within the limits [described below, without the addition of any material other than that incident to the process of manufacture] *prescribed in these specifications.*

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for Lithopone (A.S.T.M. Designation: D 208 - 26):[2]

Section 3 (*b*).—Change the requirement for zinc oxide from its present value of "2.0 per cent max." to read "1.0 per cent max."

Revision submitted, June, 1936.

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part II.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 749 (1931); also 1937 Book of A.S.T.M. Tentative Standards, p. 491.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part I, p. 835 (1928); also 1937 Book of A.S.T.M. Tentative Standards, p. 616.

[5] See p. 838.

Tentative Revision[1] of Standard Specifications for Perilla Oil, Raw or Refined (A.S.T.M. Designation: D 125 – 23):[2]

Section 1.—Change the requirement for color of perilla oil to read as follows by the addition of the italicized words:

Color.................................Not darker than a freshly prepared solution of 1.0 g. potassium dichromate in 100 cc. pure H_2SO_4 (sp. gr. 1.84), *or its equivalent in iron-cobalt solution, or in Lovibond glasses.*

Revision submitted, June, 1937.

Add to Section 3 the following requirement:

Appearance...clear and transparent at 65 C.

Revision submitted, June, 1937.

Section 3 (h).—Change the test for determining color of perilla oil from its present form to read as follows:

(*h*) *Color.*—Prepare a fresh solution of pure potassium bichromate in pure colorless H_2SO_4 (sp. gr. 1.84). This solution should be in the proportion of 1.0 g. of potassium bichromate to 100 ml. (184.0 g.) of H_2SO_4. Place the oil and colored solution in separate thin-walled, clear glass tubes of the same diameter (1 to 2 cm.) to a depth of not less than 2.5 cm. and compare the depths of color by looking transversely through the columns of liquid by transmitted light.

The iron-cobalt liquid equivalent to the above bichromate solution may be prepared as follows: Mix 29.4 ml. of Solution No. 1 with 17.6 ml. of Solution No. 2 and 53.0 ml. of Solution No. 3. The three base solutions referred to have the following composition:

Solution No. 1	$FeCl_3 \cdot 6H_2O$	5.0 g.
	HCl (2 per cent)	1.2 g.
Solution No. 2	$CoCl_2 \cdot 6H_2O$	1.0 g.
	HCl (2 per cent)	3.0 g.
Solution No. 3	Aqueous solution of HCl (2 per cent)	

The Lovibond equivalent to the above iron-cobalt solution shall be as follows: red 17.5, yellow 120.0, and blue 0.20.

The depth of liquid shall be 10.5 mm. Revision submitted, June, 1937.

Add the following test for appearance of perilla oil to this section as a new Paragraph (*i*):

(*i*) *Appearance.*—If the oil is cloudy when cold, it shall become clear and transparent when heated at 65 C. for 5 min. Revision submitted, June, 1937.

Tentative Revision[1] of Standard Specifications for Raw Tung Oil (A.S.T.M. Designation: D 12 – 33):[2]

Section 1.—In the table of properties and tests change the requirement for specific gravity from its present form: namely,

	MAXIMUM	MINIMUM
Specific Gravity at 15.5°/15.5° C.	0.943	0.9400

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part II.

to read as follows:

	MAXIMUM	MINIMUM
Specific Gravity at 15.5/15.5 C.	0.943	0.940[a]

[a] For American grown tung oil the minimum specific gravity may be as low as 0.938.

Revision submitted, June, 1935.

Change the table in Section 1 by the addition of the following requirements for color and appearance of raw tung oil:

Color	Not darker than a freshly prepared solution of 1.0 g. potassium dichromate in 100 ml. pure H_2SO_4 (sp. gr. 1.84), or its equivalent in iron-cobalt solution, or in Lovibond glasses.
Appearance	clear and transparent at 65 C.

Revision submitted, June, 1937.

Section 3.—Add the following note at the end of Paragraph (*c*) of this section:

NOTE.—If tung oil of low acid value and pale color is desired, the maximum acid value should be reduced to 3.0, and the maximum color to 0.03 g. of dichromate per 100 ml. of H_2SO_4 (or iron-cobalt, or Lovibond equivalents). Revision submitted, June, 1937.

Change the descriptions of the heating test and the quality test described in Paragraphs (*h*) and (*i*), of Section 3 from their present form to read as follows:

(*h*) *Heating Test.*—The test tubes for the oil shall be 15 cm. by 16 mm., with a mark near the bottom to indicate 5 ml. Each tube shall be closed by a cork so perforated that a glass rod 3 mm. in diameter may be moved freely. The tubes shall each weigh 14.5 ± 2.0 g. (The purpose of this requirement is to control the wall thickness.)

Fill a 1000-ml. tall-form glass beaker (height 19 cm., diameter about 9 cm.) with cottonseed oil or other suitable oil to a height of 11 cm. Place the thermometer as shown in Fig. 1 so that the bottom will be level with the bottoms of the test tubes.

Use a nitrogen-filled, chemical thermometer, engraved stem, total length 4 to 4½ in., graduated from 210 to 310 C. in 2 C. intervals; the length between 210 and 310 C. not less than 2½ in. Thermometer glass shall be well annealed. This thermometer may be suspended in the bath by a wire or may be sealed to a glass rod as shown in Fig. 1.

When the bath temperature is 293 C. (560 F.) and very slowly rising at this point, place the tube containing 5 ml. of the oil to be tested and the tube containing 5 ml. of a control sample of known value into the large holes of the cover. Note the time. Remove the source of heat for about 45 sec. and then re-apply. Before 2 min. have elapsed the temperature of the bath will have fallen to 282.2 C. (540 F.), at which point it should be kept as steady as possible. When the samples have been in the bath 9 min. raise the glass rods at intervals of 15 sec. Note the time when each sample becomes firmly set. At this period the oil will be so stiff that the entire tube may be lifted by aid of the rod. As setting or jellying takes place within a few seconds of fluidity, a good end point is afforded. Remove the samples. Heat the bath again to 293 C. (560 F.), and repeat the experiment with fresh portions of the sample.

No stirrer is used in the bath. A screen around the bath enables the temperature to be reached more easily. When the bath oil has become tarry and viscid, it should be renewed; otherwise heating may be irregular.

(*i*) *Quality Test.*[1]—The apparatus required for this test consists of an ordinary vitreous enameled iron casserole having a bottom diameter of 7.5 cm. (3 in.); a wide flange tripod with a 7.5-cm. (3-in.) opening (the object of the flange is to prevent super-heating of the sides of the casserole); a total-immersion chemical thermometer, range 30 to 300 C. and 14 to 15 in. in length.

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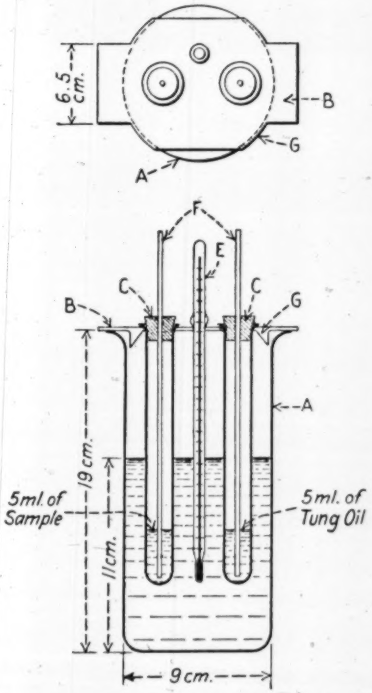
[1] Based on Worstall test.

Put into the casserole 150 g. of the oil and heat so that the temperature reaches 282.2 C. (540 F.) in 4 min. ±30 sec., stirring with the thermometer. Turn down the flame and hold the heat as near 282.2 C. ±1 C. (540 F. ± 2 F.) as possible, stirring with the thermometer, until on lifting the latter, the oil drops with a pronounced string, showing that polymerization has started. The time required after reaching 282.2 C. (540 F. ± 2 F.) until the string is noted, is the time of the heat test. For pure tung oils this will not exceed 8 min. As soon as the oil strings, remove the lamp and the thermometer, and stir with a stiff spatula until the oil is solid. After stringing, a pure tung oil will require not over 40 sec. more to become solid. When solid, allow to stand just 1 min., then turn out, upside down, on clean paper and cut at once with a clean spatula.



A. 1000-ml. tall-form beaker, 9 cm. in diameter by 19 cm. in height.
B. Support plate about 6.5 cm. wide, made of Monel metal, aluminum or stainless steel.
C. Corks.
D. Test tubes 15 cm. by 16 mm.
E. Thermometer, range 210 to 310 C.
F. Glass rods 3 mm. in diameter.
G. Guide to prevent cover from slipping.

FIG. 1.—Tung Oil Heating Test Apparatus (Revised, 1935).

Pure tung oil gives a gel that is dry, not adhering to the spatula when cut, that is firm, crumbling under pressure of the spatula without sticking, and the cut portions should crumble under pressure like dry bread crumbs. Adulterated tung oil gives a gel that is soft, sticky, and which will not crumble.

Revision submitted, June, 1935.

Add to Section 3 as a new Paragraph (*j*) a description of the test for determining color identical with that recommended above for inclusion in Section 3 (*h*) of Specifications D 125 – 23. At the end of this new Paragraph (*j*) add the same Note as proposed above for inclusion in Paragraph (*c*).

Add to Section 3 as a new Paragraph (*k*) a description of the test for appearance identical with that recommended above in Section 3 (*i*) of Specifications D 125 – 23.

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Methods of Sampling and Testing Shellac (A.S.T.M. Designation: D 29 – 33):[2]

Section 19.—Delete this section which describes the procedure for the determination of wax in orange, button, seed, and regular bleached shellac.

Revision submitted, June, 1936.

Tentative Revision[1] of Standard Methods of Testing Oleo-Resinous Varnishes (A.S.T.M. Designation: D 154 – 28):[2]

The Tentative Methods of Testing Oleo-Resinous Varnishes (Skinning Test, Alkali Resistance, Acid Number (D 154 – 35 T)[3] are in effect a tentative revision of and are intended to replace when adopted the present Standard Methods D 154 – 28.

Revision submitted, June, 1935.

Tentative Revision[1] of Standard Method of Routine Analysis of Dry Cuprous Oxide (A.S.T.M. Designation: D 283 – 36):[2]

The Tentative Method of Routine Analysis of Dry Cuprous Oxide (D 283 – 37 T)[4] is in effect a tentative revision of and is intended to replace when adopted the present Standard Method D 283 – 36.

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88 – 36):[2]

Section 4 (*c*).—Change the note following this section to read as follows by the addition of the italicized sentence:

NOTE.—The plunger commonly supplied with the viscosimeter shall never be used on instruments maintained as standards. *In case the flash point is less than 50 F. above the temperature of test, the wetting operation should be omitted in order to avoid an increase in viscosity due to evaporation of a portion of the light fraction.*

Revision submitted, June, 1937.

Section 4 (*d*).—After the first sentence add a new sentence to read as follows:

In case the flash point is less than 50 F. (10 C.) above the temperature of test, the oil shall be heated to room temperature only.

Revision submitted, June, 1937.

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part II.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 887 (1935); also 1937 Book of A.S.T.M. Tentative Standards, p. 688.

[4] See p. 855.

Tentative Revision[1] of Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (A.S.T.M. Designation: D 61 – 24):[2]

Section 1.—Change Paragraph (*b*) of this section to read as follows by the addition of the italicized word and figures and the omission of those in brackets:

(*b*) The methods herein described are not applicable to pitches having a [melting] *softening* point above [77 C. (170.6 F.)] *80 C. (176 F.)*.

Change Paragraph (*c*) of Section 1 to read as follows by the addition of the italicized figures and the omission of those in brackets:

(*c*) For the purpose of shortening the time required for testing, hard pitches having a softening point between 43 and [77] *80* C. (109.4 and [170.6] *176* F.) are cooled at 15.5 C. (60 F.) instead of at 4 C. (39.2 F.) as prescribed for soft pitches.

Revision submitted, October, 1937.

Change the center heading "A" under "Procedure" to read as follows by the addition of the italicized figures and the omission of those in brackets:

(A) Pitches Having Softening Points Between 43 and [77] *80* C. (109.4 and [170.6] *176* F.).

Revision submitted, October, 1937.

Tentative Revision[1] of Standard Specifications for Broken Slag for Waterbound Base and Wearing Course (A.S.T.M. Designation: D 65 – 23):[2]

Section 4.—Omit this section, reading as follows, renumbering the subsequent sections accordingly:

4. The percentage of wear shall be not more than 12.0 per cent.

Revision submitted, June, 1924.

Section 6.—Change the last line of this section to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

[Retained on] *Passing* 2½-in. screen . . . not [less] *more* than [85] *15* per cent.

Revision submitted, June, 1924.

Section 7 (*b*).—Omit this paragraph which refers to the method for determining the percentage of wear.

Revision submitted, June, 1924.

Tentative Revision[1] of Standard Specifications for Shovel-Run or Crusher-Run Broken Slag for Waterbound Base (A.S.T.M. Designation: D 66 – 23):[2]

Section 4.—Omit this section, reading as follows, renumbering the subsequent sections accordingly:

4. The percentage of wear shall be not more than 15.0 per cent.

Revision submitted, June, 1924.

Section 6 (*b*).—Omit this paragraph which refers to the method for determining the percentage of wear.

Revision submitted, June, 1924.

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part II.

Tentative Revision[1] of Standard Specifications for Block for Granite Block Pavements (A.S.T.M. Designation: D 59 - 26):[2]

Section 3 (a).—Change to read as follows by the addition of the italicized words:

3. (*a*) The percentage of wear and the French coefficient of wear shall be determined in accordance with the Standard Method of Test for Abrasion of Rock (A.S.T.M. Designation: D 2) of the American Society for Testing Materials, ***except that the sample shall be prepared by use of a press and the fragments shall be as uniform and as nearly cubical as practicable.***

Revision submitted, June, 1929.

Tentative Revision[1] of Standard Specifications for Block for Recut Granite Block Pavements (A.S.T.M. Designation: D 131 - 23):[2]

Section 3 (a).—Make the same change in this section as recommended in Section 3 (*a*) of Specifications D 59 - 26 above.

Revision submitted, June, 1929.

Tentative Revision[1] of Standard Specifications for Block for Durax Granite Pavements (A.S.T.M. Designation: D 132 - 23):[2]

Section 3 (a).—Make the same change in this section as recommended in Section 3 (*a*) of Specifications D 59 - 26 above.

Revision submitted, June, 1929.

Tentative Revision[1] of Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39 - 36):[2]

Section 11.—Change this section from its present form: namely,

11. *Raveled Strip Method.*—This test shall be made in accordance with the directions for the grab method (Section 10) with the following exceptions: The specimens shall be $1\frac{1}{4}$ in. in width if there are 50 or more yarns per inch, and $1\frac{1}{2}$ in. in width if there are less than 50 yarns per inch. Each specimen shall be raveled to 1 in. in width by taking from each side approximately the same number of yarns. The clamps used on the testing machine shall have faces measuring 1 by $1\frac{1}{2}$ in. or more, the longer dimension being perpendicular to the direction of application of the load.

to read as follows:

11. *Raveled Strip Method.*—This test shall be made in accordance with the directions for the grab method (Section 10) with the following exceptions: This method is applicable only to fabrics containing 50 or more yarns per inch. Each specimen shall be $1\frac{1}{4}$ in. in width and shall be raveled to 1 in. in width by taking from each side approximately the same number of yarns. The clamps used on the testing machine shall have faces measuring 1 by $1\frac{1}{2}$ in. or more, the longer dimension being perpendicular to the direction of application of the load. Revision submitted, June, 1937.

New Sections.—Add the following paragraphs at the end of these methods as new Sections 16 and 17:

16. *Twist, Warp and Filling Yarns:* (*a*) *Test Specimens.*—Specimens of the fabric shall measure at least 30 in. in the warp direction for the determination of the

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part II.

warp twist, and at least 30 in. in the filling direction, or the full width of the fabric, for the determination of the filling twist. In all operations care must be exercised to avoid any change in the twist of the yarn to be tested. The yarns may be removed from the fabric in the following manner:

Holding the yarn to be tested at one end, remove it from the fabric for a distance of approximately 10 in. (cutting the cross yarn, if necessary) and grip it at this point in one clamp of the twist tester. Remove the yarn for an additional 10 in., straighten it by applying just sufficient tension to remove the crimp (Section 8) and then grip it in the other clamp of the twist tester. The distance between clamps shall be 10 in.

(*b*) *Procedure.*—The twist of the specimen prepared as directed in Paragraph (*a*) shall be determined by one of the following procedures of the American Society for Testing Materials:

Cotton Yarn.—Section 12 of the Standard General Methods of Testing and Tolerances for Cotton Yarns and Threads (A.S.T.M. Designation: D 180).[1]

Woolen Yarn.—Section 12 of the Standard Methods of Testing and Tolerances for Woolen Yarns (A.S.T.M. Designation: D 403).[1]

Worsted Yarn.—Section 12 of the Standard Methods of Testing and Tolerances for Worsted Yarns (A.S.T.M. Designation: D 404).[1]

(*c*) *Number of Tests.*—One twist test on each of ten or more different warp yarns shall be made and the average of these ten or more tests shall be the average twist of the warp yarns. One twist test on each of ten or more different filling yarns, taken from locations in the fabric as far apart as practicable, shall be made and the average of these ten or more tests shall be the average twist of the filling yarn.

17. *Bow.*—The fabric shall be laid out smooth on a horizontal surface without tension. The position of a filling yarn in the fabric shall be marked with a line drawn on the fabric across its full width, or by other suitable means. (Marking is not necessary when distinctive colored picks are woven into the fabric at regular intervals.) A straight edge shall be placed across the fabric between the ends of this line at the edges of the fabric. The greatest distance between the line and the straight edge, measured parallel to the edges of the fabric, and the width of the fabric along the straight edge shall be measured. Similar measurements shall be made in at least three different places along a roll or piece, preferably at points 10 yd. from each end and in the center. The bow shall be expressed as a percentage of the width of the fabric and shall be the average for the three or more sets of measurements as calculated from the following formula:

$$\text{Bow, per cent} = \frac{d}{w} \times 100$$

where d = the greatest distance in inches between the marked filling yarn and the straight edge, and

w = the width of the fabric in inches.

Revision submitted, June, 1937.

Tentative Revision[2] of Standard Methods of Testing and Tolerances for Tire Cord, Woven and on Cones (A.S.T.M. Designation: D 179 - 33):[1]

Section 26.—Change the last paragraph from its present form: namely,

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[1] 1936 Book of A.S.T.M. Standards, Part II.

[2] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

A record shall be made of deviations from the scale or chart readings and corresponding corrections shall be made on the machine.

to read as follows:

When a machine is found to be in a satisfactory mechanical condition, meets the sensitivity requirements of Section 25 and then shows deviations between weights applied and dial readings, the following is considered a valid method of correction: Construct an empirical dial, the points on which shall be located by the position of the machine pointer after application of various weights as prescribed in this section.

Revision submitted, June, 1935.

Tentative Revision[1] of Standard Methods of Testing Electrical Porcelain (A.S.T.M. Designation: D 116 – 34):[2]

Section 1.—Change this section and the Note from their present form: namely,

1. These methods are intended to apply to porcelain and other vitrified ceramic materials used in the manufacture of electrical insulating parts.

NOTE.—Tests on porcelain are made on unglazed specimens since it is desired to determine the strength of the porcelain structure only. Tests have indicated, however, that the glaze may have an effect on the mechanical strength. In some cases the values obtained are higher and in other cases lower than obtained on unglazed specimens.

to read as follows:

1. These methods outline procedures for making the various tests described when they are required for an investigation or examination of porcelain or similar vitrified ceramic materials used as electrical insulating mediums. They are not to be interpreted as forming test specifications for complete insulators.

NOTE.—The glaze on a porcelain object may affect certain properties of the material, depending not only upon its presence but also upon its thickness and composition. While the test methods have been written with the intent of applying to unglazed specimens, they are equally suitable for glazed specimens. The report of test should in all cases indicate whether the specimens are glazed or unglazed.

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation: D 117 – 36):[2]

New Section.—Add the following as new Section 10, renumbering the remaining sections accordingly:

10. *Color.*—The test to determine color shall be made in accordance with the Tentative Method of Test for Color of Lubricating Oils by Means of A.S.T.M. Union Colorimeter (A.S.T.M. Designation: D 155 – 34 T) of the American Society for Testing Materials.[3]

Revision submitted, June, 1935.

Tentative Revision[1] of Standard Methods of Test for Resistivity of Insulating Materials (A.S.T.M. Designation: D 257 – 33):[2]

The Tentative Methods of Test for Insulation Resistance of Electrical

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part II.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 885 (1934); also 1937 Book of A.S.T.M. Tentative Standards, p. 710.

Insulating Materials (D 257 – 37 T)[1] are in effect a revision of and are intended to replace when adopted the present Standard Methods D 257 – 33.

Revision submitted, June, 1937.

Tentative Revision[2] of Standard Method of Sampling Coal (A.S.T.M. Designation: D 21 – 16):[3]

Section 5.—Change the last sentence of the first paragraph of this section to read as follows by the addition of the italicized words:

In the hand method samples of the quantities indicated in Table I shall be crushed so that no pieces of coal and impurities will be greater in any dimension, as judged by eye, than specified for the sample before division into two approximately equal parts.

Revision submitted, June, 1937.

Section 6 (c).—Change the second sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

The laboratory sample [s] shall include [all of one of the quarters, or] all of two opposite quarters (Fig. 34), [as may be required] *and shall not be less than 15 lb. in quantity.*

Revision submitted, June, 1937.

Section 7.—Change this section from its present form: namely,

7. Only such mechanical means as will give equally representative samples shall be used in substitution for the hand method of preparation herein standardized.

to read as follows:

7. *Mechanical Preparation.*—(*a*) Gross samples of coal containing pieces ⅜ in. and larger shall be crushed before dividing so that at least 95 per cent by weight will pass through a 4760-micron (No. 4) sieve and all will pass through a ⅜-in. round-hole screen.

Gross samples of coal all passing through a ⅜-in. round-hole screen and less than 95 per cent by weight passing through a 4760-micron (No. 4) sieve may be divided before crushing to not less than 60 lb. as described in Paragraph (*b*) by means of a riffle sampler or its equivalent.

(*b*) The entire gross sample crushed as specified under Paragraph (*a*) shall be reduced in quantity to not less than 30 lb. by passing through a riffle sampler or its equivalent. The riffle sampler shall have openings of not less than ¾ in. and not more than 1 in.

(*c*) The sample of not less than 30 lb. obtained as specified under Paragraph (*b*) shall be crushed so that all will pass through a 4760-micron (No. 4) sieve and shall then be divided by passing through a riffle with openings not less than ½ in. and not more than ¾ in., obtaining a sample of not less than 15 lb.

(*d*) The sample of not less than 15 lb. which has been crushed to all pass a 4760-micron (No. 4) sieve shall be further crushed so that 95 per cent or more by weight will pass through a 2380-micron (No. 8) sieve. The sample shall then be divided by passing through a riffle sampler with openings not over ½ in., obtaining a sample of not less than 1¾ lb. by no more than three passes through the riffle.

It is not practical to crush wet coal to pass a 2380-micron (No. 8) sieve. If the sample of not less than 15 lb., which has been crushed to pass a 4760-micron (No. 4) sieve, appears wet it shall be air-dried before crushing it so that 95 per cent passes through a 2380-micron (No. 8) sieve. However, if the determination of total moisture is of any significance, the entire sample of not less than 15 lb. of wet coal shall be placed in an air-tight container for transmission to the laboratory.

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[1] See p. 1016.

[2] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[3] 1936 Book of A.S.T.M. Standards, Part II.

(*e*) The riffle samplers specified in Paragraphs (*b*), (*c*), and (*d*) shall be similar in type to those shown in Fig. 2 (*a*) and (*b*) of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271) of the American Society for Testing Materials.[2]

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271 – 37):[2]

Section 1.—Change the quantity of the laboratory sample prescribed in Paragraphs (*e*) and (*g*) of this section from "10 lb." to read "15 lb."

Revision submitted, June, 1937.

Section 2 (*f*).—Change this section from its present form: namely,

> (*f*) *Large Riffle Sampler.*—Same as in Section 1 (*g*) for reducing coke passing the 4760-micron (No. 4) sieve to 10 lb. (see Fig. 2 (*a*)).

to read as follows:

> (*f*) *Large Riffle Sampler.*—The same as in Section 1 (*g*) (see Fig. 2 (*a*)).

Revision submitted, June, 1937.

Section 3 (*a*).—Change the first sentence in this section from its present form: namely,

> If the sample is coarser than will pass completely a 4760-micron (No. 4) sieve and larger in amount than 10 lb., quickly crush it with the jaw crusher to pass a 4760-micron (No. 4) sieve and reduce it on the larger riffle sampler to 10 lb.;[a] then crush it at once to pass an 840-micron (No. 20) sieve by passing through rolls or an enclosed grinder, and take, without sieving, a 50-g. total moisture sample, immediately after the material has passed through the crushing apparatus.

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[a] If the sample is crushed to pass a 3360-micron (No. 6) sieve it may be reduced to 5 lb.

to read as follows:

> The sample which has been collected and reduced in accordance with the Standard Method of Sampling Coal (A.S.T.M. Designation: D 21) of the American Society for Testing Materials,[2] shall be crushed[a] to pass an 840-micron (No. 20) sieve by passing through rolls or an enclosed grinder and a 50-g. total moisture sample taken, without sieving, immediately after the material has passed through the crushing apparatus.

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[a] Samples crushed to pass a 4760-micron (No. 4) sieve prepared in accordance with the Standard Method of Sampling Coal (A.S.T.M. Designation: D 21) of the American Society for Testing Materials[2] may be crushed to pass a 2380-micron (No. 8) sieve and reduced to not less than 1¾ lb. in accordance with Section 7 (*d*) of Standard Method D 21.[2]

Revision submitted, June, 1937.

Section 5 (*c*).—Insert a footnote reference "a" after the word "sample" in the first sentence of this section, this footnote to read the same as footnote "a" given above in Section 3 (*a*).

Revision submitted, June, 1937.

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1937 Supplement to Book of A.S.T.M. Standards, p. 153.

Section 43.—In Part B of this section change the tolerances for volatile matter from their present form: namely,

	Permissible Differences	
3. Volatile Matter:	Same Laboratory	Different Laboratories
Bituminous coals	0.5	1.0
Lignites	1.0	2.0
Cokes	0.2	0.4

to read as follows:

3. Volatile Matter:		
Anthracite and coke	0.2	0.4
Semianthracite, bituminous coal, subbituminous coal, and chars	0.5	1.0
Lignite and peat	1.0	2.0

Revision submitted, June, 1937.

Tentative Revision[1] of Standard Definitions of Terms Relating to Coal and Coke (A.S.T.M. Designation: D 121 - 30):[2]

The Tentative Definition of the Term Coke (D 121 - 30 T)[3] is intended to be added when adopted to the present Standard Definitions D 121 - 30.

Revision submitted, June, 1921; Modified, 1924, 1926, 1930.

Tentative Revision[1] of Standard Definitions of Terms Relating to Timber Preservatives (A.S.T.M. Designation: D 324 - 33):[2]

The following definition of the term "water-gas tar" is intended when adopted to be added to the Standard Definitions D 324 - 33:

Water-Gas Tar.—Tar produced in the manufacture of carbureted water gas by the decomposition of petroleum oil by heat in the presence of flue gas.

Revision submitted, June, 1933.

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[1] Criticisms of this revision are solicited and should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

[2] 1936 Book of A.S.T.M. Standards, Part II.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 1147 (1930); also 1937 Book of A.S.T.M. Tentative Standards, p. 648.

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SUBJECT INDEX

VOLUME 37, PART I

A

Abrasion.
  Report on Methods of Testing Oxide Coatings on Aluminum. Report of Committee B-7, Appendix II, 261.
  Tentative Methods of Test for:
    Abrasion of Coarse Aggregate by Use of the Los Angeles Machine, 801.
    Abrasion of Gravel by Use of the Deval Machine, 921.
    Abrasion Resistance of Rubber Compounds, 1134.
  Tentative Specifications for Paving Brick, 740.

Absorption Test.
  Tentative Methods of Testing Rubber Insulated Wire and Cable, 1166.
  Tentative Methods of Testing Sheet and Plate Materials Used in Electrical Insulation, 1027.
  Tentative Specifications and Methods of Test for Concrete Units for Non-Load-Bearing Masonry, 752.
  Tentative Specifications for:
    Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound, 1070.
    Insulated Wire and Cable: Heat-Resisting Rubber Compound, 1096.
    Insulated Wire and Cable: Performance Rubber Compound, 1109.

Accelerated Tests.
  See **Abrasion; Aging Tests; Corrosion; Deterioration Test; Weathering.**

Accuracy.
  Round-Table Discussion on Precision and Accuracy. Summary of Proceedings of the Fortieth Annual Meeting, 3.

Acidity.
  See **Neutralization.**

Adhesion Test.
  Tentative Methods of Testing Films Deposited from Bituminous Emulsions, 955.
  Tentative Methods of Testing Rubber Hose, 1153.

Admiralty Metal.
  Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock, 684.

Aggregates.
  Concrete and Concrete Aggregates. Report of Committee C-9, 311.
  Tentative Methods of Test for:
    Abrasion of Coarse Aggregate by Use of the Los Angeles Machine, 801.
    Abrasion of Gravel by Use of the Deval Machine, 921.
    Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate, 804.
  Tentative Specifications for:
    Concrete Aggregates, 792.
    Lightweight Aggregate for Concrete, 798.
    Standard Sizes of Coarse Aggregates for Highway Construction, 918.

Aging Tests.
  Tentative Method for Air Pressure Heat Test of Vulcanized Rubber, 1122.
  Tentative Methods of Testing Rubber Insulated Wire and Cable, 1166.
  Tentative Specifications for:
    Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound, 1070.
    Insulated Wire and Cable: Heat-Resisting Rubber Compound, 1096.
    Insulated Wire and Cable: Performance Rubber Compound, 1109.
  Yield Point of Structural Steel. Report of Research Committee, 87. Discussion, 101.

Alkalinity.
See **Neutralization.**

Alloy Steel.
See also **Steel.**
Effect of Temperature on the Properties of Metals. Report of Joint Research Committee, 164.
Iron-Chromium, Iron-Chromium-Nickel and Related Alloys. Report of Committee A-10, 153.
Report of the Wire Test Committee. Report of Committee A-5, 117. Appendix, 122.
Report on Long-Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel and 0.35 per cent Carbon Steel. Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix III, 178.
Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials, 1290.
Tentative Revision of Standard Specifications for Alloy-Steel Bolting Material for High-Temperature Service, 1306.
Tentative Specifications for:
  Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures, 616.
  Carbon-Silicon-Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels, 565.
  Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels, 572.
  Low-Carbon-Nickel Steel Plates for Boilers and Other Pressure Vessels, 577.
  Molybdenum-Steel Plates for Boilers and Other Pressure Vessels, 582.
  Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F., 622.
  Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F., 633.
  Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes, 601.
  Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service, 608.

Yield Strengths Corresponding to Small Percentages of Set. Report of Committee E-1, Appendix II, 523.

Alloys, Non-Ferrous.
See also **Aluminum; Brass; Bronze; Copper; Electrical Resistance and Heating Alloys; Magnesium; Zinc.**
Chemical Analysis of Metals. Report of Committee E-3, 543.
Copper and Copper Alloy Wires for Electrical Conductors. Report of Committee B-1, 193.
Copper and Copper Alloys, Cast and Wrought. Report of Committee B-5, 208.
Corrosion of Non-Ferrous Metals and Alloys. Report of Committee B-3, 201.
Die-Cast Metals and Alloys. Report of Committee B-6, 211.
Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys. Report of Committee B-4, 203. Discussion, 207.
Fatigue of Metals. Report of the Research Committee, 157.
Light Metals and Alloys, Cast and Wrought. Report of Committee B-7, 255.
Modulus of Elasticity of Aluminum Alloys, by R. L. Templin and Sam Tour. Report of Committee B-7, Appendix I, 258.
Nomenclature for Various Ranges in Stress in Fatigue. Report of Research Committee on Fatigue of Metals, Appendix, 159. Discussion, 162.
Non-Ferrous Metals and Alloys. Report of Committee B-2, 198.
Report on Methods of Testing Oxide Coatings on Aluminum. Report of Committee B-7, Appendix II, 261.
Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals. Report of Committee E-1, Appendix I, 518.
Spectrographic Analysis. Report of Committee E-2, 528.
Yield Strengths Corresponding to Small Percentages of Set. Report of Committee E-1, Appendix II, 523.

Aluminum.
- Light Metals and Alloys, Cast and Wrought. Report of Committee B-7, 255.
- Modulus of Elasticity of Aluminum Alloys, by R. L. Templin and Sam Tour. Report of Committee B-7, Appendix I, 258.
- Report on Methods of Testing Oxide Coatings on Aluminum. Report of Committee B-7, Appendix II, 261.
- Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
- Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.
- Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials, 1290.
- Tentative Specifications for:
  - Aluminum-Base Alloy Die Castings, 705.
  - Aluminum-Base Alloy Permanent Mold Castings, 674.
  - Aluminum-Base Alloy Sand Castings, 678.
  - Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings, 671.
  - Aluminum-Base Alloys in Ingot Form for Sand Castings, 668.

Analyses.
- See **Chemical Methods of Analyses; Testing, Physical Methods of.**
- (Methods of analyses, testing, etc., are also indexed under the materials and subjects covered by them.)

Analysis Test Data.
- See **Data, Presentation and Interpretation of.**

Annual Meeting.
- Annual Report of the Executive Committee, 54.
- Eleventh Award of Charles B. Dudley Medal. Summary of the Proceedings of the Fortieth Annual Meeting, 21.
- Fuels of Today and Tomorrow. Annual Address by the President, Arno C. Fieldner, 31.
- Summary of the Proceedings of the Fortieth Annual Meeting, 1.

Anodic Oxidation.
- Report on Methods of Testing Oxide Coatings on Aluminum. Report of Committee B-7, Appendix II, 261.

Anthracite.
- See **Coal.**

Anti-Fouling Paints.
- Tentative Method of Routine Analysis of Dry Cuprous Oxide, 855.

Antimony.
- Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
- Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.

Apparatus.
- See **Testing Apparatus.**

Arc-Welding Electrodes.
- Tentative Specifications for Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods), 589.

Arsenical Copper.
- Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock, 684.

Asbestos.
- Tentative Definitions and Terms Relating to Textile Materials, 1243.
- Tentative Methods of Testing Sheet and Plate Materials Used in Electrical Insulation, 1027.
- Textile Materials. Report of Committee D-13, 481.

Asphalt.
- Bituminous Waterproofing and Roofing Materials. Report of Committee D-8, 413.
- Road and Paving Materials. Report of Committee D-4, 395.
- Tentative Methods of Test for:
  - Asphalt Roll Roofing, Cap Sheets, and Shingles, 957.
  - Coarse Particles in Mixtures of Asphalt and Mineral Matter, 953.
  - Solid Filling and Treating Compounds Used for Electrical Insulation, 988.

Asphalt (*Continued*):
  Tentative Specifications for:
    Asphalt Cap Sheet Surfaced with Coarse Mineral Granules, 924.
    Asphalt for Damp-proofing and Waterproofing, 927.
    Asphalt for Use in Constructing Built-Up Roof Coverings, 930.
    Asphalt Roofing Surfaced with Coarse Mineral Granules, 933.
    Asphalt Roofing Surfaced with Fine Mineral Granules, 937.
    Asphalt Roofing Surfaced with Powdered Talc or Mica, 941.
    Asphalt Shingles Surfaced with Coarse Mineral Granules, 945.

Atmospheric Exposure.
  See **Corrosion; Weathering.**

Autoclave Test.
  Procedure for Making Autoclave Tests of Portland Cement Used as Basis of Comparison in Current Cooperative Studies. Report of Committee C-1, Appendix I, 290.
  Proposed Tentative Specifications and Methods of Test for Blended Cements. Report of Committee C-1, Appendix II, 291.
  Tentative Specifications for Glazed Building Units, 736.

B

Barbed Wire.
  Report of the Wire Test Committee. Report of Committee A-5, 117. Appendix, 122.
  Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Barbed Wire, 643.

Bars.
  Tentative Revision of Standard Specifications for Staybolt Wrought Iron, Hollow Rolled, 1310.
  Tentative Revision of Standard Specifications for Staybolt Wrought Iron, Solid, 1308.
  Tentative Specifications for:
    Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures, 616.
    Rolled Wrought-Iron Shapes and Bars, 664.
    Single and Double Refined Wrought-Iron Bars, 649.
    Wrought-Iron Rivets and Rivet Rounds, 659.

Bend Testing.
  Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound, 1070.
  Tentative Specifications for Rolled Zinc, 698.

Bismuth.
  Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals. Report of Committee E-1, Appendix I, 518.
  Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
  Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.

Bituminous Coal.
  See **Coal.**

Bituminous Materials.
  See **Asphalt; Petroleum Products; Timber Preservatives; Waterproofing.**

Blended Cements.
  Cement. Report of Committee C-1, 273.
  Proposed Tentative Specifications and Methods of Test for Blended Cements. Report of Committee C-1, Appendix II, 291.

Block.
  Tentative Revisions of Standard Specifications for:
    Block for Durax Granite Pavements, 1322.
    Block for Granite Block Pavements, 1322.
    Block for Recut Granite Block Pavements, 1322.
  Tentative Specifications and Methods of Test for Concrete Units for Non-Load-Bearing Masonry, 752.

Boiler Feedwater.
  Research. Report of Committee E-9, 549.

Boiler Materials.
  Tentative Revisions of Standard Specifications for:
    Boiler and Firebox Steel for Locomotives, 1303.

High Tensile Strength Carbon-Steel Plates for Fusion-Welded Pressure Vessels (Plates over 2 in. to 4 in., Inclusive, in Thickness), 1304.
High Tensile Strength Carbon-Steel Plates for Pressure Vessels (Plates 2 in. and Under in Thickness), 1303.
Tentative Specifications for:
Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures, 616.
Carbon-Silicon-Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels, 565.
Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels, 572.
Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock, 684.
Low-Carbon-Nickel Steel Plates for Boilers and Other Pressure Vessels, 577.
Molybdenum-Steel Plates for Boilers and Other Pressure Vessels, 582.
Rolled Zinc, 698.
Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F., 622.
Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F., 633.
Seamless Cold-drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes, 601.
Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service, 608.
Single and Double Refined Wrought-Iron Bars, 649.
Wrought-Iron Rivets and Rivet Rounds, 659.

Boiling Point Viscosity Number.
Diesel-Fuel-Oil Classification. Report of Committee D-2, Appendix IV, 385.

Bolting.
Tentative Revision of Standard Specifications for Alloy-Steel Bolting Material for High-Temperature Service, 1306.
Tentative Specifications for Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures, 616.

Bomb Test.
See **Aging Test.**

Braid.
Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound, 1070.

Brass.
Brass Die Castings, by J. C. Fox. Report of Committee B-6, Appendix I, 215.
Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials, 1290.
Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock, 684.

Brick.
Brick. Report of Committee C-3, 300.
Refractories. Report of Committee C-8, 307.
Tentative Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes, 785.
Tentative Specifications for:
Building Brick (Made from Clay or Shale), 733.
Glazed Building Units, 736.
Paving Brick, 740.
Sewer Brick (Made from Clay or Shale), 749.

Bronze.
Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials, 1290.
Tentative Revision of Standard Specifications for Manganese-Bronze Ingots for Sand Castings, 1310.
Tentative Revision of Standard Specifications for Manganese-Bronze Sand Castings, 1311.
Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock, 684.
Yield Strengths Corresponding to Small Percentages of Set. Report of Committee E-1, Appendix II, 523.

Building Brick.
See **Brick.**

Building Tile.
See **Tile.**

C

Cable.
Tentative Methods of Testing Rubber Insulated Wire and Cable, 1166.
Tentative Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation, 988.
Tentative Specifications for:
  Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound, 1070.
  Insulated Wire and Cable: Heat-Resisting Rubber Compound, 1096.
  Insulated Wire and Cable: Performance Rubber Compound, 1109.

Cadmium.
Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.

Calcium Chloride.
Tentative Revision of Standard Specifications for Curing Portland-Cement Concrete Slabs by Surface Application of Calcium Chloride, 1313.
Tentative Revision of Standard Specifications for Curing Portland-Cement Concrete Slabs with Calcium-Chloride Admixture, 1312.

Cap Sheets.
Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles, 957.

Carpets.
Tentative Methods of Testing Pile Floor Covering, 1235.

Castings.
See **Alloy Steel; Aluminum; Brass; Bronze; Cast Iron; Copper; Die Castings; Malleable Iron; Steel.**

Cast Iron.
Cast Iron. Report of Committee A-3, 109.
Malleable Iron Castings. Report of Committee A-7, 151.

Caustic Soda.
Proposed Methods of Chemical Analysis of Special Detergents. Report of Committee D-12, Appendix II, 477.
Tentative Specifications for Caustic Soda, 1260.

Cement.
Cement Reference Laboratory. Report of Subcommittee, Committee C-1, 288.
Cement. Report of Committee C-1, 273.
Fineness. Report of Working Committee, Committee C-1, 280.
Methods of Chemical Analysis of Portland Cement. Report of Working Committee, Committee C-1, 284.
Plastic Mortar Tests. Report of Working Committee, Committee C-1, 282.
Procedure for Making Autoclave Tests of Portland Cement Used as Basis of Comparison in Current Cooperative Studies. Report of Committee C-1, Appendix I, 290.
  Proposed Tentative Specifications and Methods of Test for Blended Cements. Report of Committee C-1, Appendix II, 291.
Tentative Method of Chemical Analysis of Portland Cement, 717.
Tentative Method of Test for Compressive Strength of Portland-Cement Mortars, 726.

Cetane Number.
Diesel-Fuel-Oil Classification. Report of Committee D-2, Appendix IV, 385.

C. F. R. Engine.
Tentative Method of Test for Knock Characteristics of Motor Fuels, 879.

Chain.
Tentative Revision of Standard Specifications for Iron and Steel Chain, 1307.

Chemical Methods of Analyses.
(Methods of chemical analysis of the ferrous and non-ferrous metals and alloys are issued collectively in a separate Society publication "A.S.T.M. Methods of Chemical Analyses of Metals, 1936.")
Chemical Analysis of Metals. Report of Committee E-3, 543.
Methods of Analysis of Paint Materials. Report of Subcommittee VIII, Committee D-1, 353.
Methods of Chemical Analysis of Portland Cement. Report of Working Committee, Committee C-1, 284.

Proposed Methods of Chemical Analysis of Special Detergents. Reports of Committee D-12, Appendix II, 477.
Proposed Methods of Chemical Analysis of Sulfonated (Sulfated) Oils. Report of Committee D-12, Appendix I, 457.
Spectrographic Analysis. Report of Committee E-2, 528.
Tentative Method of Test for Volumetric Determination of Small Amounts of Copper in Textiles, 1240.
Tentative Methods for the Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles, 1174.
Tentative Methods of Chemical Analysis of:
  Dry Cuprous Oxide, 855.
  Portland Cement, 717.
  Refractory Materials, 760.
  Soaps and Soap Products, 1266.
  Zinc Yellow Pigment (Zinc Chromate Yellow), 849.
Tentative Methods of Testing:
  Asphalt Roll Roofing, Cap Sheets, and Shingles, 957.
  Certain Wool and Part Wool Fabrics, 1231.
  Rayon, 1202.
  Wool Felt, 1226.

Chromium-Nickel Alloys.
Effect of Temperature on the Properties of Metals. Report of Joint Research Committee, 164.
Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys. Report of Committee B-4, 203. Discussion, 207.
Iron-Chromium, Iron-Chromium-Nickel and Related Alloys. Report of Committee A-10, 153.
Report on Long-Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel and 0.35 per cent Carbon Steel. Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix III, 178.
Tentative Specifications for Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures, 616.

Chromium Pigments.
Tentative Methods of Routine Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow), 849.

Chromium Steel.
Tentative Specifications for:
  Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels, 572.
  Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F., 622.
  Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes, 601.
  Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service, 608.

Cinder Concrete Block.
Tentative Specifications and Methods of Test for Concrete Units for Non-Load-Bearing Masonry, 752.

Cinders.
Tentative Specifications for Lightweight Aggregate for Concrete, 798.

Clay Hollow Tile.
See **Tile.**

Coal.
Classification of Coals. Report of Sectional Committee, 406.
Coal and Coke. Report of Committee D-5, 402.
Fuels of Today and Tomorrow. Annual Address by the President, Arno C. Fieldner, 31.
Tentative Methods of Test for:
  Drop Shatter Test for Coal, 820.
  Grindability of Coal by the Ball-Mill Method, 810.
  Grindability of Coal by the Hardgrove-Machine Method, 815.
  Tumbler Test for Coal, 827.
Tentative Revision of Standard Method of Sampling Coal, 1325.
Tentative Revision of Standard Methods of Laboratory Sampling and Analysis of Coal and Coke, 1326.

Coarse Particles.
Tentative Method of Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter, 953.

Coke.
Coal and Coke. Report of Committee D-5, 402.

Coke (*Continued*):
- Fuels of Today and Tomorrow. Annual Address by the President, Arno C. Fieldner, 31.
- Tentative Revision of Standard Methods of Laboratory Sampling and Analysis of Coal and Coke, 1326.

Color.
- Tentative Method of Test for Spectral Apparent Reflectivity of Paints, 869.
- Tentative Revision of Standard Methods of Testing Electrical Insulating Oils, 1324.
- Tentative Specifications for Raw Soybean Oil, 860.

Compression Testing.
- Plastic Mortar Tests. Report of Working Committee, Committee C-1, 282.
- A Study of Plastic Mortar Cubes, by H. W. Leavitt. Report of Committee C-9, Appendix, 321.
- Tentative Methods of Test for:
  - Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes, 785.
  - Compression Set of Vulcanized Rubber, 1127.
  - Compressive Strength of Portland-Cement Mortars, 726.
  - Sheet and Plate Materials Used in Electrical Insulation, 1027.
- Tentative Revision of Standard Method of Making and Storing Compression Test Specimens of Concrete in the Field, 1315.
- Tentative Specifications and Methods of Test for Concrete Units for Non-Load-Bearing Masonry, 752.

Concrete.
- Concrete and Concrete Aggregates. Report of Committee C-9, 311.
- Concrete Pipe. Report of Committee C-13, 337.
- Plastic Mortar Tests. Report of Working Committee, Committee C-1, 282.
- Road and Paving Materials. Report of Committee D-4, 395.
- A Study of Plastic Mortar Cubes, by H. W. Leavitt. Report of Committee C-9, Appendix, 321.
- Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate, 804.
- Tentative Revision of Standard Method of Making and Storing Compression Test Specimens of Concrete in the Field, 1315.
- Tentative Revisions of Standard Specifications for:
  - Concrete Sewer Pipe, 1311.
  - Curing Portland-Cement Concrete Slabs by Surface Application of Calcium Chloride, 1313.
  - Curing Portland-Cement Concrete Slabs with Calcium-Chloride Admixture, 1312.
  - Ready Mixed Concrete, 1313.
- Tentative Specifications for Concrete Aggregates, 792.
- Tentative Specifications for Lightweight Aggregate for Concrete, 798.
- Tentative Specifications and Methods of Test for Concrete Units for Non-Load-Bearing Masonry, 752.

Condenser Tubes.
- Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock, 684.
- Tentative Specifications for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes, 601.

Consistency.
- See also **Viscosity.**
- Symposium on Consistency: Critical Discussion on Present-Day Practices in Consistency Measurement. Report of Committee E-1, 512.
- Tentative Definitions of Terms Relating to Rheological Properties of Matter, 1300.

Copper.
- See also **Brass; Bronze.**
- Copper and Copper Alloys, Cast and Wrought. Report of Committee B-5, 208.
- Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
- Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.
- Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials, 1290.
- Tentative Specifications for:
  - Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock, 684.

Copper-Silicon Alloy Wire for General Purposes, 689.
Hard-Drawn Copper Alloy Wires for Electrical Conductors, 693.

Copper-Bearing Steel.
- Corrosion of Iron and Steel. Report of Committee A-5, 111.
- Report of the Wire Test Committee. Report of Committee A-5, 117. Appendix, 122.
- Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Barbed Wire, 643.
- Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing, 646.

Copper-Covered Steel.
- Report of the Wire Test Committee. Report of Committee A-5, 117. Appendix, 122.

Copper in Textiles.
- Tentative Method of Test for Volumetric Determination of Small Amounts of Copper in Textiles, 1240.

Copper Wire.
- Copper and Copper Alloy Wires for Electrical Conductors. Report of Committee B-1, 193.
- Tentative Methods of Testing Rubber Insulated Wire and Cable, 1166.
- Tentative Specifications for:
  - Copper-Silicon Alloy Wire for General Purposes, 689.
  - Hard-Drawn Copper Alloy Wires for Electrical Conductors, 693.
  - Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound, 1070.
  - Insulated Wire and Cable: Heat-Resisting Rubber Compound, 1096.
  - Insulated Wire and Cable: Performance Rubber Compound, 1109.

Corrosion.
- See also **Weathering.**
- Corrosion of Iron and Steel. Report of Committee A-5, 111.
- Corrosion of Non-Ferrous Metals and Alloys. Report of Committee B-3, 201.
- Iron-Chromium, Iron-Chromium-Nickel and Related Alloys. Report of Committee, A-10, 153.
- Report of the Wire Test Committee. Report of Committee A-5, 117. Appendix, 122.
- Research. Report of Committee E-9, 549.
- Symposium on Corrosion Testing Procedures. Report of Committee A-5, 115.

Cotton and Cotton Products.
- Tentative Definitions and Terms Relating to Textile Materials, 1243.
- Tentative Methods of Test for:
  - Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles, 1174.
  - Properties of Cotton Fibers, 1189.
  - Volumetric Determination of Small Amounts of Copper in Textiles, 1240.
- Tentative Revision of Standard Methods of Testing and Tolerances for Tire Cord, Woven and on Cones, 1323.
- Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound, 1070.
- Tentative Specifications for Insulated Wire and Cable: Performance Rubber Compound, 1109.
- Textile Materials. Report of Committee D-13, 481.

Cotton Fibers.
- Tentative General Methods of Test for Properties of Cotton Fibers, 1189.

Creep Tests.
- See also **Temperature, Effect of.**
- Discrepancies in the Load-Carrying Abilities of Carbon Steels at 850 F., by H. W. Gillett. Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix IV, 187. Discussion, 192.
- Effect of Temperature on the Properties of Metals. Report of Joint Research Committee, 164.
- Report on Long-Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel and 0.35 per cent Carbon Steel. Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix III, 178.

Creep Tests (*Continued*):
Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals. Report of Committee E-1, Appendix I, 518.

Creosote.
Tentative Method of Test for Tar Acids in Creosote and Creosote - Coal Tar Solutions, 832.
Tentative Specifications for Creosote for Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing, 951.

Crushing Strength.
See **Compression Testing.**

Cubes.
Plastic Mortar Tests. Report of Working Committee, Committee C-1, 282.
A Study of Plastic Mortar Cubes, by H. W. Leavitt. Report of Committee C-9, Appendix, 321.

Culvert Pipe.
Concrete Pipe. Report of Committee C-13, 337.

Cupping Tests.
Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals. Report of Committee E-1, Appendix I, 518.

Cuprous Oxide.
Tentative Method of Routine Analysis of Dry Cuprous Oxide, 855.
Tentative Revision of Standard Specifications for Curing Portland-Cement Concrete Slabs by Surface Application of Calcium Chloride, 1313.
Tentative Revision of Standard Specifications for Curing Portland-Cement Concrete Slabs with Calcium-Chloride Admixture, 1312.

D

Damp-Proofing.
See **Waterproofing.**

Data, Presentation and Interpretation of.
Methods of Testing. Report of Committee E-1, 508.
A Study of Plastic Mortar Cubes, by H. W. Leavitt. Report of Committee C-9, Appendix, 321.

Definitions of Terms.
See **Nomenclature.**

Deleterious Substances.
Tentative Specifications for Concrete Aggregates, 792.

Detergents.
See **Soaps and Detergents.**

Deterioration Test.
See also **Aging Tests.**
Tentative Methods of Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids, 1148.

Deval Abrasion Test.
Tentative Method of Test for Abrasion of Gravel by Use of the Deval Machine, 921.

Die Castings.
Brass Die Castings, by J. C. Fox. Report of Committee B-6, Appendix I, 215.
Die-Cast Metals and Alloys. Report of Committee B-6, 211.
A Study of Die Design Changes for the Improvement of the Soundness and Uniformity of Test Bars, by G. L. Werley. Report of Committee B-6, Appendix II, 223.
Tentative Specifications for Aluminum-Base Alloy Die Castings, 705.
Tentative Specifications for Magnesium-Base Alloy Die Castings, 708.

Dielectric Strength.
Report on Methods of Testing Oxide Coatings on Aluminum. Report of Committee B-7, Appendix II, 261.
Significance of Tests of Electrical Insulating Materials. Report of Committee D-9, Appendix II, 436.
Tentative Methods of Testing:
  Flexible Varnished Tubing Used for Electrical Insulation, 1056.
  Pin-Type, Lime Glass Insulators, 1008.
  Solid Filling and Treating Compounds Used for Electrical Insulation, 988.
  Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation, 1047.
Tentative Specifications for Rubber Gloves for Electrical Workers on Apparatus or Circuits not Exceeding 3000 Volts to Ground, 1062.

Diesel Fuel Oil.
Diesel-Fuel-Oil Classification. Report of Committee D-2, Appendix IV, 385.

Distillation.
Tentative Method of Test for Distillation of Plant Spray Oils, 875.

Dropping Point Test.
Proposed Method of Test for Dropping Point of Lubricating Greases. Report of Committee D-2, Appendix I, 373.

Ductility Test.
Tentative Specifications for Rolled Zinc, 698.

Dudley Medal.
Eleventh Award of Charles B. Dudley Medal. Summary of the Proceedings of the Fortieth Annual Meeting, 21.

E

Effect of Temperature.
See **Temperature, Effect of.**

Elasticity.
Modulus of Elasticity of Aluminum Alloys, by R. L. Templin and Sam Tour. Report of Committee B-7, Appendix I, 258.

Electrical Flashover Test.
Tentative Method of Testing Pin-Type, Lime Glass Insulators, 1008.

Electrical Insulating Materials.
See **Insulating Materials.**

Electrical Resistance and Heating Alloys.
Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys. Report of Committee B-4, 203. Discussion, 207.
Tentative Method of Test for Flexivity of Thermoflex (Thermostatic Metals), 711.
Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials, 1290.

Emulsions.
Tentative Methods of Testing Films Deposited from Bituminous Emulsions, 955.

Executive Committee.
Annual Report of the Executive Committee, 54.

Exhibit, Testing Apparatus.
Summary of Proceedings of the Fortieth Annual Meeting, 1.

Exposure Tests.
See **Corrosion; Weathering.**

Extraction, Methods of.
Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles, 957.

F

Fatigue.
Fatigue of Metals. Report of the Research Committee, 157.
Nomenclature for Various Ranges in Stress in Fatigue. Report of Research Committee on Fatigue of Metals, Appendix, 159. Discussion, 162.
Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals. Report of Committee E-1, Appendix I, 518.

Felt.
Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles, 957.
Tentative Methods of Testing Wool Felt, 1226.
Tentative Specifications for:
  Asphalt Roofing Surfaced with Coarse Mineral Granules, 933.
  Asphalt Roofing Surfaced with Fine Mineral Granules, 937.
  Asphalt Roofing Surfaced with Powdered Talc or Mica, 941.
  Asphalt Shingles Surfaced with Coarse Mineral Granules, 945.

Fencing.
Report of the Wire Test Committee. Report of Committee A-5, 117. Appendix, 122.
Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Barbed Wire, 643.
Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing, 646.

Ferro-Alloys.
Chemical Analysis of Metals. Report of Committee E-3, 543.

Ferrule Stock.
Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock, 684.

Fibers.
- Tentative Methods of Test for:
  - Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles, 1174.
  - Properties of Cotton Fibers, 1189.
  - Rayon, 1202.

Filler Metal.
- Tentative Specifications for Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods), 589.

Finances.
- Annual Report of the Executive Committee, 54.
- Effect of Temperature on the Properties of Metals. Report of Joint Research Committee, 164.

Fineness Modulus.
- Tentative Specifications for Concrete Aggregates, 792.

Fineness Test.
- Fineness. Report of Working Committee, Committee C-1, 280.
- Tentative General Methods of Test for Properties of Cotton Fibers, 1189.
- Tentative Specifications and Methods of Test for Fineness of Wool Tops, 1218.

Firebox Plate.
- Tentative Revisions of Standard Specifications for:
  - Boiler and Firebox Steel for Locomotives, 1303.
  - High Tensile Strength Carbon-Steel Plates for Fusion-Welded Pressure Vessels (Plates over 2 in. to 4 in., Inclusive, in Thickness), 1304.
  - High Tensile Strength Carbon-Steel Plates for Pressure Vessels (Plates 2 in. and Under in Thickness), 1303.
- Tentative Specifications for:
  - Carbon-Silicon-Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels, 565.
  - Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels, 572.
  - Low-Carbon-Nickel Steel Plates for Boilers and Other Pressure Vessels, 577.
  - Molybdenum-Steel Plates for Boilers and Other Pressure Vessels, 582.

Fire Brick.
- See **Refractories.**

Fire Tests.
- Fire Tests of Materials and Construction. Report of Committee C-5, 305.
- Tentative Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation, 1056.
- Tentative Specifications for Fire-Retardant Properties of Wood for Scaffolding and Shoring, 756.

Flanges.
- Tentative Specifications for:
  - Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures, 616.
  - Carbon-Silicon-Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels, 565.
  - Low-Carbon-Nickel Steel Plates for Boilers and Other Pressure Vessels, 577.
  - Molybdenum-Steel Plates for Boilers and Other Pressure Vessels, 582.

Flexivity Test.
- Tentative Method of Test for Flexivity of Thermoflex (Thermostatic Metals), 711.

Flexure Testing.
- Tentative Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes, 785.
- Tentative Methods of Testing Sheet and Plate Materials Used in Electrical Insulation, 1027.

Floor Coverings.
- Tentative Methods of Testing Pile Floor Covering, 1235.

Floor Tile.
- See **Tile.**

Flow Test.
- Tentative Methods of Testing Films Deposited from Bituminous Emulsions, 955.

Foots Determination.
- Tentative Specifications for Raw Soybean Oil, 860.

Forgings.
Tentative Revision of Standard Specifications for Wrought-Iron Rolled or Forged Blooms and Forgings, 1308.

Friability Test.
Tentative Method of Drop Shatter Test for Coal, 820.
Tentative Method of Tumbler Test for Coal, 827.

Fuel Oil.
Diesel-Fuel-Oil Classification. Report of Committee D-2, Appendix IV, 385.
Fuels of Today and Tomorrow. Annual Address by the President, Arno C. Fieldner, 31.
Tentative Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity, 911.
Tentative Methods of Test for Kinematic Viscosity, 900.
Tentative Standard Viscosity-Temperature Charts for Liquid Petroleum Products, 914.

Fuels.
See **Coal; Coke; Gaseous Fuels; Petroleum Products.**

G

Galvanizing.
Corrosion of Iron and Steel. Report of Committee A-5, 111.
Report of the Wire Test Committee. Report of Committee A-5, 117. Appendix, 122.
Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Barbed Wire, 643.
Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing, 646.

Gaseous Fuels.
Fuels of Today and Tomorrow. Annual Address by the President, Arno C. Fieldner, 31.
Gaseous Fuels. Report of Committee D-3, 391.

Gasoline.
Fuels of Today and Tomorrow. Annual Address by the President, Arno C. Fieldner, 31.
Natural Gasoline. Report of Subcommittee XXII, Committee D-2, 369.
Tentative Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity, 911.
Tentative Methods of Test for:
  Kinematic Viscosity, 900.
  Knock Characteristics of Motor Fuels, 879.
  Vapor Pressure of Petroleum Products (Reid Method), 888.
Tentative Specifications for Gasoline, 872.
Tentative Standard Viscosity-Temperature Charts for Liquid Petroleum Products, 914.

Gas-Welding Rods.
Tentative Specifications for Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods), 589.

Geer Oven Test.
See **Aging Test.**

Glass.
Tentative Method of Testing Pin-Type, Lime Glass Insulators, 1008.

Glazed Building Units.
Tentative Specifications for Glazed Building Units, 736.

Gloves.
Tentative Specifications for Rubber Gloves for Electrical Workers on Apparatus or Circuits not Exceeding 3000 Volts to Ground, 1062.

Gold.
Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.

Grain Size.
See **Metallography.**

Granite Block.
See **Block; Stone.**

Granular Mineral Surfacing.
See **Mineral Surfacing Material.**

Gravel.
- Tentative Methods of Test for:
  - Abrasion of Coarse Aggregate by Use of the Los Angeles Machine, 801.
  - Abrasion of Gravel by Use of the Deval Machine, 921.
  - Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate, 804.
- Tentative Specifications for Concrete Aggregates, 792.
- Tentative Specifications for Standard Sizes of Coarse Aggregates for Highway Construction, 918.

Gray Iron.
- See **Cast Iron.**

Grease.
- Proposed Method of Test for Dropping Point of Lubricating Greases. Report of Committee D-2, Appendix I, 373.

Grindability Test.
- Tentative Method of Test for Grindability of Coal by the Ball-Mill Method, 810.
- Tentative Method of Test for Grindability of Coal by the Hardgrove-Machine Method, 815.

Gypsum.
- Gypsum. Report of Committee C-11, 335.

H

Hardness Testing.
- Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals. Report of Committee E-1, Appendix I, 518.
- Tentative Methods of Testing Sheet and Plate Materials Used in Electrical Insulation, 1027.

Heat-Resistant Alloys.
- See **Electrical Resistance and Heating Alloys.**

Heat Treatment.
- Tentative Specifications for:
  - Aluminum-Base Alloy Permanent Mold Castings, 674.
  - Aluminum-Base Alloy Sand Castings, 678.
  - Carbon-Silicon-Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels, 565.

Hiding Power.
- Methods of Analysis of Paint Materials. Report of Subcommittee VIII, Committee D-1, 353.
- Tentative Method of Test for Relative Dry Hiding Power of Paints, 866.
- Tentative Method of Test for Relative Dry Hiding Power of White Pigments, 844.

High Temperature Materials and Tests.
- Discrepancies in the Load-Carrying Abilities of Carbon Steels at 850 F., by H. W. Gillett. Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix IV, 187. Discussion, 192.
- Effect of Temperature on the Properties of Metals. Report of Joint Research Committee, 164.
- Proposed Revision of Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 34 T). Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix I, 173.
- Report on Long-Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel and 0.35 per cent Carbon Steel. Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix III, 178.
- Tentative Method of Test for Flexivity of Thermoflex (Thermostatic Metals), 711.
- Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials, 1290.
- Tentative Revision of Standard Specifications for Alloy-Steel Bolting Material for High-Temperature Service, 1306.
- Tentative Specifications for:
  - Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures, 616.
  - Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F., 622.

Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F., 633.
Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes, 601.
Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service, 608.

Hollow Tile.
See **Tile.**

Honorary Membership Awards.
Award of Honorary Memberships. Summary of Proceedings of the Fortieth Annual Meeting, 3.

Hose.
Tentative Methods of Testing Rubber Hose, 1153.

Hydrostatic Test.
Tentative Methods of Testing Rubber Hose, 1153.

I

Ignition Quality.
Diesel-Fuel-Oil Classification. Report of Committee D-2, Appendix IV, 385.

Immaturity Count.
Tentative General Methods of Test for Properties of Cotton Fibers, 1189.

Immersion Tests.
Corrosion of Iron and Steel. Report of Committee A-5, 111.

Impact Test.
Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals. Report of Committee E-1, Appendix I, 518.
Significance of Tests of Electrical Insulating Materials. Report of Committee D-9, Appendix II, 436.
A Study of Die Design Changes for the Improvement of the Soundness and Uniformity of Test Bars, by G. L. Werley. Report of Committee B-6, Appendix II, 223.
Tentative Method of Drop Shatter Test for Coal, 820.

Industrial Waters.
See **Boiler Feedwater; Water.**

Insulating Materials.
Electrical Insulating Materials. Report of Committee D-9, 424.
Proposed Revisions in Standards for Electrical Insulating Materials. Report of Committee D-9, Appendix I, 430.
Significance of Tests of Electrical Insulating Materials. Report of Committee D-9, Appendix II, 436.
Tentative Methods of Testing:
  Flexible Varnished Tubing Used for Electrical Insulation, 1056.
  Insulation Resistance of Electrical Insulating Materials, 1016.
  Pin-Type, Lime Glass Insulators, 1008.
  Rubber Insulated Wire and Cable, 1166.
  Sheet and Plate Materials Used in Electrical Insulation, 1027.
  Shellac Used for Electrical Insulation, 1041.
  Solid Filling and Treating Compounds Used for Electrical Insulation, 988.
  Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation, 1047.
Tentative Revision of Standard Methods of Testing Electrical Insulating Oils, 1324.
Tentative Revision of Standard Methods of Testing Electrical Porcelain, 1324.
Tentative Specifications for:
  Black Bias-Cut Varnished Cloth Tape Used for Electrical Insulation, 979.
  Flexible Varnished Tubing Used in Electrical Insulation, 985.
  Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound, 1070.
  Insulated Wire and Cable: Heat-Resisting Rubber Compound, 1096.
  Insulated Wire and Cable: Performance Rubber Compound, 1109.
  Phenolic Laminated Sheet for Radio Applications, 973.
  Rubber Gloves for Electrical Workers on Apparatus or Circuits not Exceeding 3000 Volts to Ground, 1062.

Iodine Number.
Tentative Specifications for Raw Soybean Oil, 860.

Iron.
See also **Alloy Steel; Cast Iron; Malleable Iron; Steel; Wrought Iron.**
Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.

Iron-Chromium-Nickel Alloys.
See **Alloy Steel; Chromium-Nickel Alloys.**

K

Kinematic Viscosity.
Method for Converting Kinematic Viscosity to Saybolt Universal Viscosity, by C. E. Headington and J. C. Geniesse. Report of Committee D-2, Appendix III, 380.
Tentative Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity, 911.
Tentative Methods of Test for Kinematic Viscosity, 900.
Tentative Standard Viscosity-Temperature Charts for Liquid Petroleum Products, 914.

Knock Characteristics.
Tentative Method of Test for Knock Characteristics of Motor Fuels, 879.

L

Laboratory Inspection.
Cement Reference Laboratory. Report of Subcommittee, Committee C-1, 288.

Lacquers.
Paint, Varnish, Lacquer, and Related Products. Report of Committee D-1, 339.

Lead.
Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.

Lead-Coated Steel.
Report of the Wire Test Committee. Report of Committee A-5, 117. Appendix, 122.

Life Tests.
See **Aging Test.**

Light Metals.
See **Aluminum; Magnesium.**

Lightweight Aggregates.
Tentative Specifications for Lightweight Aggregate for Concrete, 798.

Lithopone.
Tentative Method of Test for Relative Dry Hiding Power of White Pigments, 844.
Tentative Revision of Standard Specifications for Lithopone, 1316.

Los Angeles Abrasion Test.
Tentative Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine, 801.

Lubricants.
See **Petroleum Products.**

M

Magnesium.
Light Metals and Alloys, Cast and Wrought. Report of Committee B-7, 255.
Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.
Tentative Specifications for Magnesium-Base Alloy Die Castings, 708.

Magnesium Pigments.
Tentative Specifications for Titanium-Magnesium Pigment, 840.
Tentative Specifications for Zinc Sulfide Magnesium Pigment, 842.

Magnesium Sulfate Test.
Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate, 804.

Malleable Iron.
Malleable Iron Castings. Report of Committee A-7, 151.

Manganese.
Methods of Chemical Analysis of Portland Cement. Report of Working Committee, Committee C-1, 284.

Manganese Bronze.
Tentative Revision of Standard Specifications for Manganese-Bronze Ingots for Sand Castings, 1310.

Tentative Revision of Standard Specifications for Manganese-Bronze Sand Castings, 1311.

Masonry Building Units.
See also **Brick; Tile.**
Brick. Report of Committee C-3, 300.
Hollow Masonry Building Units. Report of Committee C-10, 333.
Tentative Specifications and Methods of Test for Concrete Units for Non-Load-Bearing Masonry, 752.

Mechanical Analysis.
Tentative Methods of Test for:
  Coarse Particles in Mixtures of Asphalt and Mineral Matter, 953.
  Drop Shatter Test for Coal, 820.
  Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles, 965.
  Sieve Analysis of Non-Granular Mineral Surfacing for Asphalt Roofing and Shingles, 969.
  Tumbler Test for Coal, 827.
Tentative Specifications for Concrete Aggregates, 792.
Tentative Specifications for Standard Sizes of Coarse Aggregates for Highway Construction, 918.

Medal Award.
Eleventh Award of Charles B. Dudley Medal. Summary of the Proceedings of the Fortieth Annual Meeting, 21.

Membership.
Annual Report of the Executive Committee, 54.

Metallography.
Metallography. Report of Committee E-4, 546.
Report on Methods of Testing Oxide Coatings on Aluminum. Report of Committee B-7, Appendix II, 261.
A Study of Die Design Changes for the Improvement of the Soundness and Uniformity of Test Bars, by G. L. Werley. Report of Committee B-6, Appendix II, 223.

Methods of Testing.
See **Testing, Physical Methods of.**

Mica.
Tentative Method of Test for Sieve Analysis of Non-Granular Mineral Surfacing for Asphalt Roofing and Shingles, 969.

Microscopic Tests.
Report on Methods of Testing Oxide Coatings on Aluminum. Report of Committee B-7, Appendix II, 261.
Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals. Report of Committee E-1, Appendix I, 518.
Tentative General Methods of Test for Properties of Cotton Fibers, 1189.
Tentative Methods for the Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles, 1174.
Tentative Methods of Testing and Tolerances for Rayon, 1202.
Tentative Specifications and Methods of Test for Fineness of Wool Tops, 1218.

Mineral Surfacing Material.
Tentative Method of Test for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles, 965.
Tentative Method of Test for Sieve Analysis of Non-Granular Mineral Surfacing for Asphalt Roofing and Shingles, 969.

Mobility.
Tentative Definitions of Terms Relating to Rheological Properties of Matter, 1300.

Modified Soda.
Proposed Methods of Chemical Analysis of Special Detergents. Report of Committee D-12, Appendix II, 477.
Tentative Specifications for Modified Soda (Sesquicarbonate Type), 1261.

Modulus of Elasticity.
Modulus of Elasticity of Aluminum Alloys, by R. L. Templin and Sam Tour. Report of Committee B-7, Appendix I, 258.
Yield Strengths Corresponding to Small Percentages of Set. Report of Committee E-1, Appendix II, 523.

Molded Insulating Materials.
See **Insulating Materials.**

Molybdenum Steel.
Tentative Specifications for:
  Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures, 616.

Molybdenum Steel (*Continued*):
  Molybdenum-Steel Plates for Boilers and Other Pressure Vessels, 582.
  Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F., 622.
  Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F., 633.
  Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes, 601.
  Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service, 608.

Mortars.
  Plastic Mortar Tests. Report of Working Committee, Committee C-1, 282.
  A Study of Plastic Mortar Cubes, by H. W. Leavitt. Report of Committee C-9, Appendix, 321.
  Tentative Method of Test for Compressive Strength of Portland-Cement Mortars, 726.

Motor Fuel.
  See **Fuel Oil; Gasoline; Petroleum Products.**

Motor Lubricants.
  See **Petroleum Products.**

Muntz Metal.
  Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock, 684.

N

Naval Stores.
  Acid and Saponification Number and Unsaponifiable Matter. Report of Subcommittee IV, Committee D-17, 503.
  Naval Stores. Report of Committee D-17, 501.
  Tentative Method of Test for Acid Number of Rosin, 835.
  Tentative Method of Test for Saponification Number of Rosin, 836.

Neutralization.
  Acid and Saponification Number and Unsaponifiable Matter. Report of Subcommittee IV, Committee D-17, 503.
  Tentative Method of Test for Acid Number of Rosin, 835.
  Tentative Specifications for Raw Soybean Oil, 860.

Nickel.
  Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
  Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.

Nickel Steel.
  See also **Alloy Steel; Chromium-Nickel Alloys.**
  Tentative Specifications for Low-Carbon-Nickel Steel Plates for Boilers and Other Pressure Vessels, 577.
  Tentative Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F., 622.

Nomenclature.
  Nomenclature for Various Ranges in Stress in Fatigue. Report of Research Committee on Fatigue of Metals, Appendix, 159. Discussion, 162.
  Tentative Definitions of Terms Relating to:
    Refractories, 790.
    Rheological Properties of Matter, 1300.
    Soaps and Detergents, 1289.
    Textile Materials, 1243.
  Tentative revision of definition of water-gas tar, Standard Definitions of Terms Relating to Timber Preservatives, 1327.

O

Octane Number.
  Tentative Method of Test for Knock Characteristics of Motor Fuels, 879.

Oils.
  See **Creosote; Fuel Oil; Paint Vehicles; Petroleum Products; Transformer Oils.**

Ostwald Viscosimeter.
  Tentative Methods of Test for Kinematic Viscosity, 900.

Oxide Coatings.
  Report on Methods of Testing Oxide Coatings on Aluminum. Report of Committee B-7, Appendix II, 261.

P

Paints.
  See also **Lacquers; Paint Vehicles; Pigments; Shellac; Varnish.**
  Accelerated Tests for Protective Coatings. Report of Subcommittee VII, Committee D-1, 350.
  Methods of Analysis of Paint Materials. Report of Subcommittee VIII, Committee D-1, 353.

Paint, Varnish, Lacquer, and Related Products. Report of Committee D-1, 339.
Symposium on Correlation Between Accelerated Laboratory Tests and Service Tests on Protective and Decorative Coatings. Report of Committee D-1, 339.
Tentative Method of Test for Relative Dry Hiding Power of Paints, 866.
Tentative Method of Test for Spectral Apparent Reflectivity of Paints, 869.

Paint Vehicles.
Tentative Revision of Standard Specifications for Perilla Oil, Raw or Refined; and for Raw Tung Oil, 1317.
Tentative Specifications for Raw Soybean Oil, 860.
Testing of Paint Vehicles. Report of Subcommittee III, Committee D-1, 347.

Papers and Publications.
Annual Report of the Executive Committee, 54.
Summary of the Proceedings of the Fortieth Annual Meeting, 1.

Paving Materials.
See **Aggregates; Asphalt; Concrete; Road Materials.**

Perilla Oil.
Tentative Revision of Standard Specifications for Perilla Oil, Raw or Refined, 1317.
Testing of Paint Vehicles. Report of Subcommittee III, Committee D-1, 347.

Permanent Mold Castings.
Tentative Specifications for Aluminum-Base Alloy Permanent Mold Castings, 674.
Tentative Specifications for Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings, 671.

Petroleum Products.
Diesel-Fuel-Oil Classification. Report of Committee D-2, Appendix IV, 385.
Fuels of Today and Tomorrow. Annual Address by the President, Arno C. Fieldner, 31.
Method for Converting Kinematic Viscosity to Saybolt Universal Viscosity, by C. E. Headington and J. C. Geniesse. Report of Committee D-2, Appendix III, 380.
Natural Gasoline. Report of Subcommittee XXII, Committee D-2, 369.
Petroleum Products and Lubricants. Report of Committee D-2, 360.
Petroleum Products and Lubricants. Report of Sectional Committee, 389.
Proposed Method of Test for Dropping Point of Lubricating Greases. Report of Committee D-2, Appendix I, 373.
Proposed Method of Test for Unsulfonated Residue of Plant Spray Oils. Report of Committee D-2, Appendix II, 375.
Tentative Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity, 911.
Tentative Methods of Test for:
  Distillation of Plant Spray Oils, 875.
  Kinematic Viscosity, 900.
  Knock Characteristics of Motor Fuels, 879.
  Vapor Pressure of Petroleum Products (Reid Method), 888.
Tentative Revision of Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter, 1320.
Tentative Specifications for Gasoline, 872.
Tentative Viscosity-Temperature Charts for Liquid Petroleum Products, 914.
Turbine Oils. Report of Subcommittee XI, Committee D-2, 367.

Phenolic Sheet.
Tentative Specifications for Phenolic Laminated Sheet for Radio Applications, 973.

Phosphorus.
Methods of Chemical Analysis of Portland Cement. Report of Working Committee, Committee C-1, 284.

Pigments.
See also **Paints.**
Methods of Analysis of Paint Materials. Report of Subcommittee VIII, Committee D-1, 353.
Paint, Varnish, Lacquer, and Related Products. Report of Committee D-1, 339.
Tentative Method of Routine Analysis of Dry Cuprous Oxide, 855.
Tentative Method of Test for Relative Dry Hiding Power of White Pigments, 844.

Pigments (*Continued*):
Tentative Methods of Routine Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow), 849.
Tentative Revisions of Standard Specifications for:
  Lithopone, 1316.
  Zinc Oxide, 1316.
  Zinc Sulfide, 1316.
Tentative Specifications for:
  Basic Sulfate White Lead, 838.
  Titanium-Magnesium Pigment, 840.
  Zinc Sulfide Magnesium Pigment, 842.
Pile Floor Covering.
Tentative Methods of Testing Pile Floor Covering, 1235.
Pine.
See **Timber.**
Pipe and Piping Materials.
See also **Tubes.**
Concrete Pipe. Report of Committee C-13, 337.
Tentative Method of Test for Short-time High-Temperature Tension Tests of Metallic Materials, 1290.
Tentative Revision of Standard Specifications for Welded Wrought-Iron Pipe, 1308.
Tentative Specifications for:
  Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock, 684.
  Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods), 589.
  Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F., 622.
  Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F., 633.
  Wrought-Iron Rivets and Rivet Rounds, 659.
Pitch.
Bituminous Waterproofing and Roofing Materials. Report of Committee D-8, 413.
Tentative Specifications for Coal-Tar Pitch for Roofing, Damp-proofing and Waterproofing, 948.
Tentative Specifications for Creosote for Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing, 951.
Plant Spray Oils.
Proposed Method of Test for Unsulfonated Residue of Plant Spray Oils. Report of Committee D-2, Appendix II, 375.
Tentative Method of Test for Distillation of Plant Spray Oils, 875.
Plasticity.
Tentative Definitions of Terms Relating to Rheological Properties of Matter, 1300.
Plastic Mortar Test.
Plastic Mortar Tests. Report of Working Committee, Committee C-1, 282.
A Study of Plastic Mortar Cubes, by H. W. Leavitt. Report of Committee C-9, Appendix, 321.
Tentative Method of Test for Compressive Strength of Portland-Cement Mortars, 726.
Plates.
See also **Sheets.**
Tentative Methods of Testing Sheet and Plate Materials Used in Electrical Insulation, 1027.
Tentative Revisions of Standard Specifications for:
  Boiler and Firebox Steel for Locomotives, 1303.
  High Tensile Strength Carbon-Steel Plates for Fusion-Welded Pressure Vessels (Plates over 2 in. to 4 in., Inclusive, in Thickness), 1304.
  High Tensile Strength Carbon-Steel Plates for Pressure Vessels (Plates 2 in. and Under in Thickness), 1303.
Tentative Specifications for:
  Carbon-Silicon-Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels, 565.
  Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels, 572.
  Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods), 589.
  Low-Carbon-Nickel Steel Plates for Boilers and Other Pressure Vessels, 577.
  Rolled Zinc, 698.
  Wrought-Iron Plates, 653.

Platinum.
Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.
Polymerization Time Test.
Tentative Methods of Testing Shellac Used for Electrical Insulation, 1041.
Porcelain.
Tentative Revision of Standard Methods of Testing Electrical Porcelain, 1324.
Portland Cement.
See **Cement.**
Pothead Compounds.
Tentative Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation, 988.
Precision.
Round-Table Discussion on Precision and Accuracy. Summary of Proceedings of the Fortieth Annual Meeting, 3.
Presidential Address.
Fuels of Today and Tomorrow. Annual Address by the President, Arno C. Fieldner, 31.
Pressure Vessel Materials.
See **Boiler Materials.**
Protective Coatings.
See **Corrosion; Galvanizing; Lacquers; Paints; Pigments; Shellac; Timber Preservatives; Varnish.**
Publications.
See **Papers and Publications.**

R

Radiographs.
A Study of Die Design Changes for the Improvement of the Soundness and Uniformity of Test Bars, by G. L. Werley. Report of Committee B-6, Appendix II, 223.
Radio Spaghetti.
Tentative Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation, 1056.
Tentative Specifications for Flexible Varnished Tubing Used in Electrical Insulation, 985.
Rattler Test.
Tentative Specifications for Paving Brick, 740.
Rayon.
Tentative Definitions and Terms Relating to Textile Materials, 1243.
Tentative Methods for the Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles, 1174.
Tentative Methods of Testing and Tolerances for Rayon, 1202.
Textile Materials. Report of Committee D-13, 481.
Ready Mixed Concrete.
Tentative Revision of Standard Specifications for Ready Mixed Concrete, 1313.
Reflectivity.
Tentative Method of Test for Spectral Apparent Reflectivity of Paints, 869.
Refractories.
Refractories. Report of Committee C-8, 307.
Tentative Definitions of Terms Relating to Refractories, 790.
Tentative Methods of Chemical Analysis of Refractory Materials, 760.
Tentative Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes, 785.
Reid Vapor Pressure Test.
Natural Gasoline. Report of Subcommittee XXII, Committee D-2, 369.
Tentative Method of Test for Vapor Pressure of Petroleum Products (Reid Method), 888.
Research.
Annual Report of the Executive Committee, 54.
Effect of Temperature on the Properties of Metals. Report of Joint Research Committee, 164.
Fatigue of Metals. Report of the Research Committee, 157.
Report on Long-Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel and 0.35 per cent Carbon Steel. Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix III, 178.
Research. Report of Committee E-9, 549.
Yield Point of Structural Steel. Report of Research Committee, 87. Discussion, 101.
Resistance Alloys.
See **Electrical Resistance and Heating Alloys.**

Resistivity Tests.
- Significance of Tests of Electrical Insulating Materials. Report of Committee D-9, Appendix II, 436.
- Tentative Methods of Test for Insulation Resistance of Electrical Insulating Materials, 1016.
- Tentative Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation, 988.

Revisions.
- (Revisions of Standards and Tentative Standards are given in the reports of the standing committees responsible and are also indexed under the subjects covered by them.)

Rivets.
- Tentative Revision of Standard Specifications for Structural Rivet Steel, 1305.
- Tentative Specifications for Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels, 572.
- Tentative Specifications for Wrought-Iron Rivets and Rivet Rounds, 659.

Road Materials.
- See also **Aggregates; Asphalt; Concrete.**
- (Items on road materials are also indexed under the materials and subjects covered by them.)
- Road and Paving Materials. Report of Committee D-4, 395.

Roofing.
- Bituminous Waterproofing and Roofing Materials. Report of Committee D-8, 413.
- Tentative Methods of Test for:
  - Asphalt Roll Roofing, Cap Sheets, and Shingles, 957.
  - Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles, 965.
  - Sieve Analysis of Non-Granular Mineral Surfacing for Asphalt Roofing and Shingles, 969.
- Tentative Specifications for:
  - Asphalt Cap Sheet Surfaced with Coarse Mineral Granules, 924.
  - Asphalt for Damp-proofing and Waterproofing, 927.
  - Asphalt for Use in Constructing Built-Up Roof Coverings, 930.
  - Asphalt Roofing Surfaced with Coarse Mineral Granules, 933.
  - Asphalt Roofing Surfaced with Fine Mineral Granules, 937.
  - Asphalt Roofing Surfaced with Powdered Talc or Mica, 941.
  - Asphalt Shingles Surfaced with Coarse Mineral Granules, 945.
  - Coal-Tar Pitch for Roofing, Damp-proofing and Waterproofing, 948.

Rosin.
- Acid and Saponification Number and Unsaponifiable Matter. Report of Subcommittee IV, Committee D-17, 503.
- Naval Stores. Report of Committee D-17, 501.
- Tentative Method of Test for Acid Number of Rosin, 835.
- Tentative Method of Test for Saponification Number of Rosin, 836.

Rubber Products.
- Rubber Products. Report of Committee D-11, 444.
- Tentative Methods of Test for:
  - Abrasion Resistance of Rubber Compounds, 1134.
  - Air Pressure Heat Test of Vulcanized Rubber, 1122.
  - Changes in Properties of Rubber and Rubber-Like Materials in Liquids, 1148.
  - Compression Set of Vulcanized Rubber, 1127.
  - Rubber Hose, 1153.
  - Rubber Insulated Wire and Cable, 1166.
  - Sheet and Plate Materials Used in Electrical Insulation, 1027.
- Tentative Specifications for:
  - Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound, 1070.
  - Insulated Wire and Cable: Heat-Resisting Rubber Compound, 1096.
  - Insulated Wire and Cable: Performance Rubber Compound, 1109.
  - Rubber Gloves for Electrical Workers on Apparatus or Circuits not Exceeding 3000 Volts to Ground, 1062.

S

Sampling.
- (Methods of sampling are indexed under the materials and subjects covered by them.)

Sand.
See also **Aggregates.**
Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate, 804.
Tentative Specifications for Concrete Aggregates, 792.

Saponification Number.
Acid and Saponification Number and Unsaponifiable Matter. Report of Subcommittee IV, Committee D-17, 503.
Tentative Method of Test for Saponification Number of Rosin, 836.
Tentative Specifications for Raw Soybean Oil, 860.

Saybolt Viscosity.
Method for Converting Kinematic Viscosity to Saybolt Universal Viscosity, by C. E. Headington and J. C. Geniesse. Report of Committee D-2, Appendix III, 380.
Tentative Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity, 911.
Tentative Revision of Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter, 1320.
Tentative Viscosity-Temperature Charts for Liquid Petroleum Products, 914.

Scaffolding.
Fire Tests of Materials and Construction. Report of Committee C-5, 305.
Tentative Specifications for Fire-Retardant Properties of Wood for Scaffolding and Shoring, 756.

Sea Water Corrosion.
See **Corrosion; Immersion Tests.**

Set Test.
Tentative Methods of Testing Rubber Hose, 1153.

Sewer Brick.
Tentative Specifications for Sewer Brick (Made from Clay or Shale), 749.

Sewer Pipe.
Tentative Revision of Standard Specifications for Concrete Sewer Pipe, 1311.

Shapes.
Tentative Specifications for Rolled Wrought-Iron Shapes and Bars, 664.

Shatter Test.
Tentative Method of Drop Shatter Test for Coal, 820.

Shear or Punching Tests.
Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals. Report of Committee E-1, Appendix I, 518.
Tentative Method of Test for Compression Set of Vulcanized Rubber, 1127.

Sheets.
See also **Plates.**
Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals. Report of Committee E-1, Appendix I, 518.
Tentative Methods of Testing Sheet and Plate Materials Used in Electrical Insulation, 1027.
Tentative Revision of Standard Specifications for Uncoated Wrought-Iron Sheets, 1310.
Tentative Revision of Standard Specifications for Zinc-Coated (Galvanized) Wrought-Iron Sheets, 1310.
Tentative Specifications for:
  Asphalt Cap Sheet Surfaced with Coarse Mineral Granules, 924.
  Phenolic Laminated Sheet for Radio Applications, 973.
  Rolled Zinc, 698.

Shellac.
Tentative Methods of Testing Shellac Used for Electrical Insulation, 1041.
Tentative Revision of Standard Methods of Sampling and Testing Shellac, 1320.

Shingles.
Bituminous Waterproofing and Roofing Materials. Report of Committee D-8, 413.
Tentative Methods of Test for:
  Asphalt Roll Roofing, Cap Sheets, and Shingles, 957.
  Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles, 965.
  Sieve Analysis of Non-Granular Mineral Surfacing for Asphalt Roofing and Shingles, 969.
Tentative Specifications for Asphalt Roofing Surfaced with Coarse Mineral Granules, 933.
Tentative Specifications for Asphalt Shingles Surfaced with Coarse Mineral Granules, 945.

Shoring.
Fire Tests of Materials and Construction. Report of Committee C-5, 305.
Tentative Specifications for Fire-Retardant Properties of Wood for Scaffolding and Shoring, 756.
Shrinkage Test.
Tentative Methods of Testing and Tolerances for Certain Wool and Part Wool Fabrics, 1231.
Sieves.
Tentative Method of Test for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles, 965.
Tentative Method of Test for Sieve Analysis of Non-Granular Mineral Surfacing for Asphalt Roofing and Shingles, 969.
Silicon Steel.
Tentative Specifications for:
  Carbon-Silicon-Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels, 565.
  Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels, 572.
  Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F., 622.
Silk.
Tentative Definitions and Terms Relating to Textile Materials, 1243.
Tentative Methods for the Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles, 1174.
Silver.
Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.
Slag.
Tentative Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine, 801.
Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate, 804.
Tentative Revision of Standard Specifications for Broken Slag for Waterbound Base and Wearing Course, 1321.
Tentative Revision of Standard Specifications for Shovel-Run or Crusher-Run Broken Slag for Waterbound Base, 1321.
Tentative Specifications for:
  Concrete Aggregates, 792.
  Lightweight Aggregate for Concrete, 798.
  Standard Sizes of Coarse Aggregate for Highway Construction, 918.
Slate.
Tentative Method of Test for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles, 965.
Soaps and Detergents.
Proposed Methods of Chemical Analysis of Special Detergents. Report of Committee D-12, Appendix II, 477.
Proposed Methods of Chemical Analysis of Sulfonated (Sulfated) Oils. Report of Committee D-12, Appendix I, 457.
Soaps and Detergents. Report of Committee D-12, 454.
Tentative Definitions of Terms Relating to Soaps and Detergents, 1289.
Tentative Methods of Sampling and Chemical Analysis of Soaps and Soap Products, 1266.
Tentative Specifications for:
  Caustic Soda, 1260.
  Milled Toilet Soap, 1264.
  Modified Soda (Sesquicarbonate Type), 1261.
  Soda Ash, 1262.
Soda Ash.
Proposed Methods of Chemical Analysis of Special Detergents. Report of Committee D-12, Appendix II, 477.
Tentative Specifications for Soda Ash, 1262.
Sodium Sulfate.
Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate, 804.
Softening Point.
Proposed Method of Test for Dropping Point of Lubricating Greases. Report of Committee D-2, Appendix I, 373.
Tentative Revision of Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method), 1321.
Soundness.
Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate, 804.

Soybean Oil.
Tentative Specifications for Raw Soybean Oil, 860.
Testing of Paint Vehicles. Report of Subcommittee III, Committee D-1, 347.

Specifications.
(Specifications are indexed under the materials and subjects covered by them.)

Spectral Apparent Reflectivity.
Tentative Method of Test for Spectral Apparent Reflectivity of Paints, 869.

Spectrographic Analysis.
Spectrographic Analysis. Report of Committee E-2, 528.
Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.

Speed of Testing, Effect of.
Yield Point of Structural Steel. Report of Research Committee, 87. Discussion, 101.

Splitting Resistance Test.
Tentative Methods of Testing Wool Felt, 1226.

Stainless Steel.
See **Alloy Steel; Chromium-Nickel Alloys.**

Standardization.
Annual Report of the Executive Committee, 54.
Cement Reference Laboratory. Report of Subcommittee, Committee C-1, 288.
Standards. Report of Committee E-10 on Standards, 555.
Summary of the Proceedings of the Fortieth Annual Meeting, 1.

Statistical Analysis.
Methods of Testing. Report of Committee E-1, 508.
A Study of Plastic Mortar Cubes, by H. W. Leavitt. Report of Committee C-9, Appendix, 321.

Staybolts.
Tentative Revision of Standard Specifications for Staybolt Wrought Iron, Hollow Rolled, 1310.
Tentative Revision of Standard Specifications for Staybolt Wrought Iron, Solid, 1308.

Steel.
Chemical Analysis of Metals. Report of Committee E-3, 543.
Corrosion of Iron and Steel. Report of Committee A-5, 111.
Discrepancies in the Load-Carrying Abilities of Carbon Steels at 850 F., by H. W. Gillett. Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix IV, 187. Discussion, 192.
Effect of Temperature on the Properties of Metals. Report of Joint Research Committee, 164.
Fatigue of Metals. Report of the Research Committee, 157.
Iron-Chromium, Iron-Chromium-Nickel and Related Alloys. Report of Committee A-10, 153.
Nomenclature for Various Ranges in Stress in Fatigue. Report of Research Committee on Fatigue of Metals, Appendix, 159. Discussion, 162.
Proposed Revision of Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 34 T). Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix I, 173.
Proposed Revision of Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 34 T). Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix II, 176.
Report of the Wire Test Committee. Report of Committee A-5, 117. Appendix, 122.
Report on Long-Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel and 0.35 per cent Carbon Steel. Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix III, 178.
Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals. Report of Committee E-1, Appendix, 518.

Steel (*Continued*):
- Steel. Report of Committee A-1, 81.
- Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials, 1290.
- Tentative Revisions of Standard Specifications for:
  - Alloy-Steel Bolting Material for High-Temperature Service, 1306.
  - Boiler and Firebox Steel for Locomotives, 1303.
  - High Tensile Strength Carbon-Steel Plates for Fusion-Welded Pressure Vessels (Plates over 2 in. to 4 in., Inclusive, in Thickness), 1304.
  - High Tensile Strength Carbon-Steel Plates for Pressure Vessels (Plates 2 in. and Under in Thickness), 1303.
  - Iron and Steel Chain, 1307.
  - Structural Rivet Steel, 1305.
- Tentative Specifications for:
  - Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures, 616.
  - Carbon-Silicon-Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels, 565.
  - Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels, 572.
  - Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods), 589.
  - Low-Carbon-Nickel Steel Plates for Boilers and Other Pressure Vessels, 577.
  - Molybdenum-Steel Plates for Boilers and Other Pressure Vessels, 582.
  - Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F., 622.
  - Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F., 633.
  - Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes, 601.
  - Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service, 608.
  - Zinc-Coated (Galvanized) Iron or Steel Barbed Wire, 643.
  - Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing, 646.
- Yield Point of Structural Steel. Report of Research Committee, 87. Discussion, 101.
- Yield Strengths Corresponding to Small Percentages of Set. Report of Committee E-1, Appendix II, 523.

Still Tubes.
- Tentative Specifications for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service, 608.

Stone.
- Tentative Methods of Test for:
  - Abrasion of Coarse Aggregate by Use of the Los Angeles Machine, 801.
  - Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles, 965.
  - Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate, 804.
- Tentative Revisions of Standard Specifications for:
  - Block for Durax Granite Pavements, 1322.
  - Block for Granite Block Pavements, 1322.
  - Block for Recut Granite Block Pavements, 1322.
- Tentative Specifications for Concrete Aggregates, 792.
- Tentative Specifications for Standard Sizes of Coarse Aggregate for Highway Construction, 918.

Strip.
- Tentative Method of Test for Flexivity of Thermoflex (Thermostatic Metals), 711.
- Tentative Specifications for Rolled Zinc, 698.

Structural Clay Tile.
- See **Tile.**

Structural Steel.
- Yield Point of Structural Steel. Report of Research Committee, 87. Discussion, 101.

Structural Wrought Iron.
- Tentative Specifications for Rolled Wrought-Iron Shapes and Bars, 664.

Sulfonated Oils.
Proposed Methods of Chemical Analysis of Sulfonated (Sulfated) Oils. Report of Committee D-12, Appendix I, 457.

Summary of Proceedings.
Summary of the Proceedings of the Fortieth Annual Meeting, 1.

Super-Pure Metals.
Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.

Suspended Level Viscosimeter.
Tentative Methods of Test for Kinematic Viscosity, 900.

T

Talc.
Tentative Method of Test for Sieve Analysis of Non-Granular Mineral Surfacing for Asphalt Roofing and Shingles, 969.

Tape.
Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation, 1047.
Tentative Specifications for Black Bias-Cut Varnished Cloth Tape Used for Electrical Insulation, 979.
Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound, 1070.

Tar.
Tentative Revision of Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method), 1321.

Tar Acids Test.
Tentative Method of Test for Tar Acids in Creosote and Creosote - Coal Tar Solutions, 832.

Temperature, Effect of.
Discrepancies in the Load-Carrying Abilities of Carbon Steels at 850 F., by H. W. Gillett. Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix IV, 187. Discussion, 192.
Effect of Temperature on the Properties of Metals. Report of Joint Research Committee, 164.
Proposed Revision of Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 34 T). Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix I, 173.
Proposed Revision of Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 34 T). Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix II, 176.
Report on Long-Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel and 0.35 per cent Carbon Steel. Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, Appendix III, 178.
Tentative Method of Test for Flexivity of Thermoflex (Thermostatic Metals), 711.
Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials, 1290.

Temper Test.
Tentative Specifications for Rolled Zinc, 698.

Tension Testing.
Modulus of Elasticity of Aluminum Alloys, by R. L. Templin and Sam Tour. Report of Committee B-7, Appendix I, 258.
Proposed Revision of Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 34 T). Report of Joint Research Committee on Effect of Temperature on Properties of Metals, Appendix I, 173.
Proposed Revision of Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 34 T). Report of Joint Research Committee on Effect of Temperature on Properties of Metals, Appendix II, 176.
Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals. Report of Committee E-1, Appendix I, 518.
A Study of Die Design Changes for the Improvement of the Soundness and Uniformity of Test Bars, by G. L. Werley. Report of Committee B-6, Appendix II, 223.

Tension Testing (*Continued*):
  Tentative Methods of Testing:
    Rubber Hose, 1153.
    Rubber Insulated Wire and Cable, 1166.
    Sheet and Plate Materials Used in Electrical Insulation, 1027.
    Short-Time High-Temperature Tension Tests of Metallic Materials, 1290.
    Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation, 1047.
  Tentative Specifications for:
    Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound, 1070.
    Insulated Wire and Cable: Heat-Resisting Rubber Compound, 1096.
    Insulated Wire and Cable: Performance Rubber Compound, 1109.
  Yield Strengths Corresponding to Small Percentages of Set. Report of Committee E-1, Appendix II, 523.

Tentative Revisions.
  (Revisions of Standards and Tentative Standards are given in the reports of the standing committees responsible and are also indexed under the materials and subjects covered by them.)
  Tentative Revisions of A.S.T.M. Standards, 1303.

Test Bars.
  A Study of Die Design Changes for the Improvement of the Soundness and Uniformity of Test Bars, by G. L. Werley. Report of Committee B-6, Appendix II, 223.

Testing Apparatus.
  See also **Testing, Physical Methods of.**
  Tentative Method of Test for Knock Characteristics of Motor Fuels, 879.
  Tentative Specifications for Rolled Zinc, 698.

Testing, Physical Methods of.
  (Methods of analysis, sampling, testing, etc., and revisions thereof, are also indexed under the materials and subjects covered by them.)
  Methods of Testing. Report of Committee E-1, 508.
  Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals. Report of Committee E-1, Appendix I, 518.
  Tentative Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine, 801.
  Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials, 1290.
  Yield Point of Structural Steel. Report of Research Committee, 87. Discussion, 101.
  Yield Strengths Corresponding to Small Percentages of Set. Report of Committee E-1, Appendix II, 523.

Textile Materials.
  Proposed Revisions in Standards for Textile Materials. Report of Committee D-13, Appendix, 491.
  Tentative Definitions and Terms Relating to Textile Materials, 1243.
  Tentative Methods of Testing:
    Asphalt Roll Roofing, Cap Sheets and Shingles, 957.
    Certain Wool and Part Wool Fabrics, 1231.
    Flexible Varnished Tubing Used for Electrical Insulation, 1056.
    Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles, 1174.
    Pile Floor Covering, 1235.
    Properties of Cotton Fibers, 1189.
    Rayon, 1202.
    Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation, 1047.
    Volumetric Determination of Small Amounts of Copper in Textiles, 1240.
    Wool Felt, 1226.
  Tentative Revision of Standard General Methods of Testing Woven Textile Fabrics, 1322.
  Tentative Revision of Standard Methods of Testing and Tolerances for Tire Cord, Woven and on Cones, 1323.
  Tentative Specifications for:
    Asphalt Roofing Surfaced with Coarse Mineral Granules, 933.
    Asphalt Roofing Surfaced with Powdered Talc or Mica, 941.

Black Bias-Cut Varnished Cloth Tape Used for Electrical Insulation, 979.
Fineness of Wool Tops, 1218.
Flexible Varnished Tubing Used in Electrical Insulation, 985.
Textile Materials. Report of Committee D-13, 481.

Thermal Shock Test.
Tentative Method of Testing Pin-Type, Lime Glass Insulators, 1008.

Thermoflex (Thermostatic Metals).
Tentative Method of Test for Flexivity of Thermoflex (Thermostatic Metals), 711.

Thermometers, A.S.T.M.
Air chamber thermometer, Tentative Method of Test for Vapor Pressure of Petroleum Products (Reid Method), 891.
Kinematic viscosity test thermometer, Tentative Methods of Test for Kinematic Viscosity, 902.
Water-bath thermometer, Tentative Method of Test for Vapor Pressure of Petroleum Products (Reid Method), 890.

Thermostatic Metals.
Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys. Report of Committee B-4, 203. Discussion, 207.

Thickness.
Report on Methods of Testing Oxide Coatings on Aluminum. Report of Committee B-7, Appendix II, 261.
Tentative Method of Testing Pin-Type, Lime Glass Insulators, 1008.
Tentative Methods of Testing Wool Felt, 1226.
Tentative Specifications for:
Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound, 1070.
Insulated Wire and Cable: Heat-Resisting Rubber Compound, 1096.
Rubber Gloves for Electrical Workers on Apparatus or Circuits not Exceeding 3000 Volts to Ground, 1062.

Tile.
Hollow Masonry Building Units. Report of Committee C-10, 333.
Tentative Specifications for Glazed Building Units, 736.

Timber.
Fire Tests of Materials and Construction. Report of Committee C-5, 305.
Tentative Specifications for Fire-Retardant Properties of Wood for Scaffolding and Shoring, 756.
Timber. Report of Committee D-7, 410.

Timber Preservatives.
Tentative Method of Test for Tar Acids in Creosote and Creosote-Coal Tar Solutions, 832.
Tentative revision of definition of water-gas tar, Standard Definitions of Terms Relating to Timber Preservatives, 1327.

Tin.
Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.

Tire Cord and Fabrics.
Tentative Methods of Test for Abrasion Resistance of Rubber Compounds, 1134.
Tentative Revision of Standard Methods of Testing and Tolerances for Tire Cord, Woven and on Cones, 1323.

Titanium Pigments.
Tentative Specifications for Titanium-Magnesium Pigment, 840.

Transformer Oils.
Tentative Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity, 911.
Tentative Methods of Test for Kinematic Viscosity, 900.
Tentative Methods of Test for Insulation Resistance of Electrical Insulating Materials, 1016.
Tentative Revision of Standard Methods of Testing Electrical Insulating Oils, 1324.

Transverse Testing.
See **Flexure Testing.**

Trolley Wire.
Copper and Copper Alloy Wires for Electrical Conductors. Report of Committee B-1, 193.

Tubes.
See also **Pipe and Piping Materials.**
Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials, 1290.
Tentative Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation, 1056.

Tubes (*Continued*):
  Tentative Specifications for:
    Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock, 684.
    Flexible Varnished Tubing Used in Electrical Insulation, 985.
    Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes, 601.
    Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service, 608.

Tumbler Test.
  Tentative Method of Tumbler Test for Coal, 827.

Tung Oil.
  Tentative Revision of Standard Specifications for Raw Tung Oil, 1317.
  Testing of Paint Vehicles. Report of Subcommittee III, Committee D-1, 347.

Turbine Oils.
  Turbine Oils. Report of Subcommittee XI, Committee D-2, 367.

Twist.
  Tentative Definitions and Terms Relating to Textile Materials, 1243.

U

Unsulfonated Residue.
  Proposed Method of Test for Unsulfonated Residue of Plant Spray Oils. Report of Committee D-2, Appendix II, 375.

V

Valves.
  Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures, 616.

Vapor Pressure.
  Natural Gasoline. Report of Subcommittee XXII, Committee D-2, 369.
  Tentative Method of Test for Vapor Pressure of Petroleum Products (Reid Method), 888.

Varnish.
  Paint, Varnish, Lacquer, and Related Products. Report of Committee D-1, 339.
  Tentative Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation, 1056.
  Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation, 1047.
  Tentative Specifications for Black Bias-Cut Varnished Cloth Tape Used for Electrical Insulation, 979.
  Tentative Specifications for Flexible Varnished Tubing Used in Electrical Insulation, 985.

Vibration Damping.
  Tentative Method of Test for Compression Set of Vulcanized Rubber, 1127.

Viscosity.
  Method for Converting Kinematic Viscosity to Saybolt Universal Viscosity, by C. E. Headington and J. C. Geniesse. Report of Committee D-2, Appendix III, 380.
  Tentative Definitions of Terms Relating to Rheological Properties of Matter, 1300.
  Tentative Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity, 911.
  Tentative Methods of Test for Kinematic Viscosity, 900.
  Tentative Revision of Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter, 1320.
  Tentative Viscosity-Temperature Charts for Liquid Petroleum Products, 914.

Viscosity-Gravity Number.
  Diesel-Fuel-Oil Classification. Report of Committee D-2, Appendix IV, 385.

Volatile Matter.
  Tentative Revision of Standard Methods of Laboratory Sampling and Analysis of Coal and Coke, 1326.

Voltage Test.
  Report on Methods of Testing Oxide Coatings on Aluminum. Report of Committee B-7, Appendix II, 261.
  Significance of Tests of Electrical Insulating Materials. Report of Committee D-9, Appendix II, 436.
  Tentative Methods of Testing Rubber Insulated Wire and Cable, 1166.
  Tentative Specifications for:
    Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound, 1070.
    Insulated Wire and Cable: Heat-Resisting Rubber Compound, 1096.
    Insulated Wire and Cable: Performance Rubber Compound, 1109.

Volume Change.
  Tentative Methods of Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids, 1148.
  Tentative Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation, 988.
Vulcanized Rubber.
  See **Rubber Products.**

W

Water.
  See also **Absorption Test.**
  Research. Report of Committee E-9, 549.
  Water for Industrial Uses. Report of Committee D-19, 505.
Water Gas Tar.
  Tentative Revision of Standard Definitions of Terms Relating to Timber Preservatives, 1327.
Waterproofing.
  Bituminous Waterproofing and Roofing Materials. Report of Committee D-8, 413.
  Tentative Methods of Testing Films Deposited from Bituminous Emulsions, 955.
  Tentative Specifications for:
    Asphalt for Damp-proofing and Waterproofing, 927.
    Asphalt for Use in Constructing Built-Up Roof Coverings, 930.
    Coal-Tar Pitch for Roofing, Damp-proofing and Waterproofing, 948.
    Creosote for Priming Coat with Coal-Tar Pitch in Damp-proofing and Waterproofing, 951.
Weathering.
  See also **Corrosion.**
  Accelerated Tests for Protective Coatings. Report of Subcommittee VII, Committee D-1, 350.
  Symposium on Correlation Between Accelerated Laboratory Tests and Service Tests on Protective and Decorative Coatings. Report of Committee D-1, 339.
  Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate, 804.
Weight.
  Tentative Methods of Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids, 1148.
Welding.
  Tentative Specifications for:
    Carbon-Silicon-Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels, 565.
    Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels, 572.
    Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods), 589.
    Low-Carbon-Nickel Steel Plates for Boilers and Other Pressure Vessels, 577.
    Molybdenum-Steel Plates for Boilers and Other Pressure Vessels, 582.

White Lead Pigments.
  Tentative Method of Test for Relative Dry Hiding Power of White Pigments, 844.
  Tentative Specifications for Basic Sulfate White Lead, 838.

Wire.
  Copper and Copper Alloy Wires for Electrical Conductors. Report of Committee B-1, 193.
  Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys. Report of Committee B-4, 203. Discussion, 207.
  Report of the Wire Test Committee. Report of Committee A-5, 117. Appendix, 122.
  Tentative Methods of Testing Rubber Insulated Wire and Cable, 1166.
  Tentative Specifications for:
    Copper-Silicon Alloy Wire for General Purposes, 689.
    Hard-Drawn Copper Alloy Wires for Electrical Conductors, 693.
    Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound, 1070.
    Insulated Wire and Cable: Heat-Resisting Rubber Compound, 1096.
    Insulated Wire and Cable: Performance Rubber Compound, 1109.
    Zinc-Coated (Galvanized) Iron or Steel Barbed Wire, 643.
    Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing, 646.

Wood.
See **Timber.**
Wool.
Tentative Definitions and Terms Relating to Textile Materials, 1243.
Tentative Methods for the Identification of Fibers in Textiles and for the Quantitative Analysis of Textiles, 1174.
Tentative Methods of Testing:
Certain Wool and Part Wool Fabrics, 1231.
Pile Floor Covering, 1235.
Wool Felt, 1226.
Tentative Specifications and Methods of Test for Fineness of Wool Tops, 1218.
Textile Materials. Report of Committee D-13, 481.
Wrought Iron.
Chemical Analysis of Metals. Report of Committee E-3, 543.
Corrosion of Iron and Steel. Report of Committee A-5, 111.
Proposed Revisions in Tentative Standards for Wrought Iron. Report of Committee A-2, Appendix, 107.
Tentative Revisions of Standard Specifications for:
Iron and Steel Chain, 1307.
Staybolt Wrought Iron, Hollow Rolled, 1310.
Staybolt Wrought Iron, Solid, 1308.
Uncoated Wrought-Iron Sheets, 1310.
Welded Wrought-Iron Pipe, 1308.
Wrought-Iron Rolled or Forged Blooms and Forgings, 1308.
Zinc-Coated (Galvanized) Wrought-Iron Sheets, 1310.
Tentative Specifications for:
Rolled Wrought-Iron Shapes and Bars, 664.
Single and Double Refined Wrought-Iron Bars, 649.
Wrought-Iron Plates, 653.
Wrought-Iron Rivets and Rivet Rounds, 659.
Wrought Iron. Report of Committee A-2, 103.

Y

Yarns.
See also **Textile Materials.**
Tentative Methods of Testing and Tolerances for Certain Wool and Part Wool Fabrics, 1231.
Tentative Methods of Testing and Tolerances for Rayon, 1202.
Yield Point.
Yield Point of Structural Steel. Report of Research Committee, 87. Discussion, 101.
Yield Strength.
Yield Strengths Corresponding to Small Percentages of Set. Report of Committee E-1, Appendix II, 523.
Yield Value.
Tentative Definitions of Terms Relating to Rheological Properties of Matter, 1300.

Z

Zinc.
Non-Ferrous Metals and Alloys. Report of Committee B-2, 198.
Report of the Wire Test Committee. Report of Committee A-5, 117. Appendix, 122.
Standards and Pure Materials. Report of Subcommittee V, Committee E-2, 531.
A Study of Die Design Changes for the Improvement of the Soundness and Uniformity of Test Bars, by G. L. Werley. Report of Committee B-6, Appendix II, 223.
Super-Purity Metals, by Thomas A. Wright. Report of Committee E-2, Appendix, 538.
Tentative Specifications for:
Rolled Zinc, 698.
Zinc-Coated (Galvanized) Iron or Steel Barbed Wire, 643.
Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing, 646.
Zinc Pigments.
Tentative Method of Test for Relative Dry Hiding Power of White Pigments, 844.
Tentative Methods of Routine Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow), 849.
Tentative Revision of Standard Specifications for Zinc Oxide, 1316.
Tentative Revision of Standard Specifications for Zinc Sulfide, 1316.
Tentative Specifications for Zinc Sulfide Magnesium Pigment, 842.

AUTHOR INDEX

VOLUME 37, PART I

A

Anderson, R. P.
Report of Committee D-2 on Petroleum Products and Lubricants, 360.

Aufderhaar, H. C.
Report of Committee A-3 on Cast Iron, 109.

B

Ball, H. J.
Report of Committee D-13 on Textile Materials, 481.

Bash, F. E.
Report of Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys, 203.

Bates, P. H.
Report of Committee C-1 on Cement, 273.
Report of Committee E-9 on Research, 549.

Battle, J. D.
Report of Sectional Committee on Classification of Coals, 406.

Bigler, H. P.
Report of Committee A-1 on Steel, 81.

Bolton, J. W.
Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, 164.

Bowman, J. J.
Report of Committee E-4 on Metallography, 546.

Boyd, T. A.
Report of Committee D-2 on Petroleum Products and Lubricants, 360.
Report of Sectional Committee on Petroleum Products and Lubricants, 389.

Bright, H. A.
Report of Committee E-3 on Chemical Analysis of Metals, 543.

C

Capp, J. A.
Report of Committee B-1 on Copper and Copper Alloy Wires for Electrical Conductors, 193.
Report of Subcommittee XI, Committee D-2, on Turbine Oils, 367.

Carpenter, Arthur W.
Report of Committee D-11 on Rubber Products, 444.

Chapman, Cloyd M.
Report of Committee E-10 on Standards, 555.

Churchill, H. V.
Report of Committee E-2 on Spectrographic Analysis, 528.

Conner, R. M.
Report of Committee D-3 on Gaseous Fuels, 391.

Crampton, D. K.
Report of Committee B-5 on Copper and Copper Alloys, Cast and Wrought, 208.

Cross, Howard C.
Report on Long-Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel and 0.35 per cent Carbon Steel. Appendix III, Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, 178.

Crum, R. W.
Report of Committee C-9 on Concrete and Concrete Aggregates, 311.

Curtis, H. L.
Significance of the Resistivity Test. Appendix II, Committee D-9, Significance of Tests of Electrical Insulating Materials, 439.

D

Davis, M. P.
Significance of the Impact Test. Appendix II, Committee D-9, Significance of Tests of Electrical Insulating Materials, 443.

E

Edwards, Junius D.
  Report on Methods of Testing Oxide Coatings on Aluminum. Appendix II, Report of Committee B-7, 261.
Emery, F. E.
  Report of Committee C-10 on Hollow Masonry Building Units, 333.

F

Faragher, P. V.
  Report of Committee B-6 on Die-Cast Metals and Alloys, 211.
Farnsworth, F. F.
  Report of Committee A-5 on Corrosion of Iron and Steel, 111.
  Report of the Wire Test Committee. Report of Committee A-5, 117.
Fieldner, Arno C.
  Annual Report of the Executive Committee, 54.
  Fuels of Today and Tomorrow. Annual Address by the President, 31.
  Report of Committee D-3 on Gaseous Fuels, 391.
  Report of Committee D-5 on Coal and Coke, 402.
  Report of Sectional Committee on Classification of Coals, 406.
Foote, J. H.
  Report of Committee B-1 on Copper and Copper Alloy Wires for Electrical Conductors, 193.
Fox, J. C.
  Brass Die Castings. Appendix I, Report of Committee B-6, 215.
French, H. J.
  Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, 164.
Fuller, T. S.
  Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, 201.
Fulweiler, W. H.
  Report of Committee E-1 on Methods of Testing, 508.

G

Galloway, T. R.
  Report of Committee A-5 on Corrosion of Iron and Steel, 111.
Gann, J. A.
  Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, 255.
Gardner, H. A.
  Report of Subcommittee III, Committee D-1, on Testing of Paint Vehicles, 347.
Geniesse, J. C.
  Method for Converting Kinematic Viscosity to Saybolt Universal Viscosity. Appendix III, Report of Committee D-2, 380.
Gillett, H. W.
  Discrepancies in the Load-Carrying Abilities of Carbon Steels at 850 F. Appendix IV, Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, 187.
Ginder, J. W.
  Report of Committee C-11 on Gypsum, 335.
Gough, H. J.
  Discussion, 192.

H

Hall, R. E.
  Report of Committee D-19 on Water for Industrial Uses, 505.
Hanna, W. C.
  Report of Working Committee, Committee C-1, on Methods of Chemical Analysis of Portland Cement, 284.
Harper, R. B.
  Report of Committee D-3 on Gaseous Fuels, 391.
Harvey, Dean.
  Report of Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys, 203.
  Significance of the Dielectric Strength Test. Appendix II, Committee D-9, Significance of Tests of Electrical Insulating Materials, 436.
  Discussion, 207.
Hayden, O. M.
  Report of Committee D-11 on Rubber Products, 444.
Headington, C. E.
  Method for Converting Kinematic Viscosity to Saybolt Universal Viscosity. Appendix III, Report of Committee D-2, 380.

Hecht, Max.
Report of Committee D-19 on Water for Industrial Uses, 505.

Hess, R. E.
Report of Sectional Committee on Petroleum Products and Lubricants, 389.

Hickson, E. F.
Report of Subcommittee VIII, Committee D-1, on Methods of Analysis of Paint Materials, 353.

Hubbard, Prévost.
Report of Committee D-4 on Road and Paving Materials, 395.
Report of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 413.

J

Jenks, G. F.
Yield Strengths Corresponding to Small Percentages of Set. Appendix II, Report of Committee E-1, 523.

K

Kelley, E. F.
Report of Committee C-13 on Concrete Pipe, 337.

Körber, Friedrich.
Discussion, 101.

L

Lang, F. C.
Report of Committee D-4 on Road and Paving Materials, 395.

Leavitt, H. W.
A Study of Plastic Mortar Cubes. Appendix, Report of Committee C-9, 321.

Litehiser, R. R.
Report of Committee C-9 on Concrete and Concrete Aggregates, 311.

Loving, M. W.
Report of Committee C-13 on Concrete Pipe, 337.

Lowther, J. G.
Report on Long-Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel and 0.35 per cent Carbon Steel. Appendix III, Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, 178.

Lundell, G. E. F.
Report of Committee E-3 on Chemical Analysis of Metals, 543.

M

Marshall, C. H.
Report on Usefulness and Limitation of Various Tests as Applied to Sheet Metals. Appendix I, Report of Committee E-1, 518.

Mathewson, C. H.
Report of Committee B-5 on Copper and Copper Alloys, Cast and Wrought, 208.

McBurney, J. W.
Report of Committee C-3 on Brick, 300.

Mehl, R. F.
Report of Committee B-2 on Non-Ferrous Metals and Alloys, 198.

Miller, Rudolph P.
Report of Committee C-5 on Fire Tests of Materials and Construction, 305.

Mochel, N. L.
Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, 164.

Moore, H. F.
Report of the Research Committee on Fatigue of Metals, 157.
Discussion, 163.

Moore, O. L.
Report of Working Committee, Committee C-1, on Plastic Mortar Tests, 282.

Morgan, H. H.
Report of Committee A-1 on Steel, 81.

N

Newell, H. D.
Report of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys, 153.

Newlin, J. A.
Report of Committee D-7 on Timber, 410.

Nitchie, C. C.
Report of Committee E-2 on Spectrographic Analysis, 528.

Norton, John T.
Report of Committee E-4 on Metallography, 546.

O

Oberfell, G. G.
Report of Subcommittee XXII, Committee D-2, on Natural Gasoline, 369.

P

Parsons, D. E.
Report of Committee C-10 on Hollow Masonry Building Units, 333.

Paul, M. Rea.
Report of Committee D-1 on Paint, Varnish, Lacquer, and Related Products, 339.

Pearson, C. J.
Report of Working Committee, Committee C-1, on Fineness, 280.

Peterson, R. E.
Discussion, 162.

Phelps, S. M.
Report of Committee C-8 on Refractories, 307.

R

Rogers, Allen.
Report of Committee D-1 on Paint, Varnish, Lacquer, and Related Products, 339.

Rother, W. H.
Report of Committee A-3 on Cast Iron, 109.

Rutan, E. J.
Report of Committee D-9 on Electrical Insulating Materials, 424.

S

Schwartz, H. A.
Report of Committee A-7 on Malleable Iron Castings, 151.

Schweim, H. J.
Report of Committee C-11 on Gypsum, 335.

Selvig, W. A.
Report of Committee D-5 on Coal and Coke, 402.

Smith, E. K.
Report of Committee A-7 on Malleable Iron Castings, 151.

Smith, P. J.
Report of Committee E-1 on Methods of Testing, 508.

Speh, C. F.
Report of Committee D-17 on Naval Stores, 501.

Strauss, Jerome.
Report of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys, 153.

Sullivan, J. D.
Report of Committee C-8 on Refractories, 307.

T

Taylor, Fitzhugh.
Report of Committee C-5 on Fire Tests of Materials and Construction, 305.

Taylor, T. Smith.
Report of Committee D-9 on Electrical Insulating Materials, 424.

Templin, R. L.
Modulus of Elasticity of Aluminum Alloys. Appendix I, Report of Committee B-7, 258.

Thum, E. E.
Report of Committee B-2 on Non-Ferrous Metals and Alloys, 198.

Tour, Sam.
Modulus of Elasticity of Aluminum Alloys. Appendix I, Report of Committee B-7, 258.
Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, 201.
Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, 255.

Townsend, J. R.
Report of Committee B-6 on Die-Cast Metals and Alloys, 211.

Trevithick, H. P.
Report of Committee D-12 on Soaps and Detergents, 454.

V

Van Schoick, E. H.
Report of Committee C-8 on Refractories, 307.

Van Zile, B. S.
Report of Committee D-12 on Soaps and Detergents, 454.

Veitch, F. P.
Report of Committee D-17 on Naval Stores, 501.

von Schrenk, Hermann.
Report of Committee D-7 on Timber, 410.

W

Walter, L. W.
Report of Committee C-1 on Cement, 273.

Warren, G. E.
Report of Subcommittee, Committee C-1, on Cement Reference Laboratory, 288.

Warwick, C. L.
Annual Report of the Executive Committee, 54.
Report of Committee E-9 on Research, 549.
Report of Committee E-10 on Standards, 555.

Webster, W. R.
Discussion, 207.

Weiss, J. M.
Report of Committee D-8 on Bituminous Waterproofing and Roofing Materials, 413.

Werley, G. L.
A Study of Die Design Changes for the Improvement of the Soundness and Uniformity of Test Bars. Appendix II, Report of Committee B-6, 223.

Whitcomb, W. H.
Report of Committee D-13 on Textile Materials, 481.

Whittemore, J. W.
Report of Committee C-3 on Brick, 300.

Withey, M. O.
Report of Research Committee on Yield Point of Structural Steel, 87.
Discussion, 102.

Woodroffe, G. H.
Report of Committee A-2 on Wrought Iron, 103.

Wright, Thomas A.
Report of Subcommittee V, Committee E-2, on Standards and Pure Materials, 531.
Super-Purity Metals. Appendix, Report of Committee E-2, 538.

Y

Young, J. B.
Report of Committee A-2 on Wrought Iron, 103.

Z

Zeigler, C. C.
Report of Subcommittee IV, Committee D-17, on Acid and Saponification Number and Unsaponifiable Matter, 503.